STUDY ON SWELLING AND CLEANING OF EGG YOLK DEPOSITS AT LABORATORY SCALE

by

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A thesis submitted to
The University of Birmingham
for the degree of
DOCTOR OF PHILOSOPHY

School of Chemical Engineering
University of Birmingham
MARCH 2019

ABSTRACT

Fouling has always been a major problem in the food processing industry where thermal treatment is involved. Cleaning of food deposit formed from fouling phenomenon is always a tough challenge for food industry as it affects product safety and hygiene, and production cost. Egg yolk was used in this study as a model fouling deposit on stainless steel surface. It mainly contains protein and lipids which makes it difficult to be cleaned once thermally induced on a surface. Experiments were conducted to study the effect of various parameters on swelling and removal of egg yolk deposits during hydration, the influence of chemical and mechanical action on swelling and removal of egg yolk deposit during hydration, and the effect of various parameters on cleaning of egg yolk deposits at laboratory scale and the adhesive and cohesive strength of egg yolk deposits.

Sets of experiments were carried out by soaking the egg yolk deposits in different kind of hydrating solutions (water, NaOH, LAS solution and commercial detergent solution) at different concentrations (0.5 and 1% w/v), temperatures (20, 40, 60, 80^{o}C), stirring speeds (200 and 400 rpm), stirring methods (magnetic stirrer and mixing impeller) and sample configurations (horizontal and vertical). Deposit swelling, moisture content and removal were measured during the experiments. Experiments were also conducted to study the effect of different temperatures (40, 60 and 80^{o}C), cleaning agents (NaOH and LAS solution), and chemical concentrations (1 and 5 % w/v) on cleaning of egg yolk deposits by cleaning rig at 0.5 ms^{-1} flow velocity. Micromanipulation technique were used in this study to measure the apparent adhesive and cohesive strength of egg yolk deposits at different pre-soaking time (15 and 30 minutes), temperatures (20, 40, and 60^{o}C), cleaning fluids (NaOH and LAS solution), and chemical concentrations (1 and 5 % w/v).

It was found that temperature plays a major role on swelling and cleaning of egg yolk deposits during hydration. In some cases, swelling and deposit removal occurred simultaneously during hydration. At, 80^{o}C hydration, a complete removal was achieved in all cases. Mechanical and chemical action was also found to affect swelling and deposit removal of egg yolk deposits during hydration. However, the effect of different sample configurations was unclear.

Cleaning of egg yolk deposits by cleaning rig can divided into three different phases i.e. lag phase, removal phase and constant phase. No complete deposit removal was achieved after 4 hours of rinsing with water, even at the highest temperature (80^{o}C). At 40^{o}C, no complete removal was obtained after 4 hours of experiment in all cases. Increasing the chemical concentration would increase the deposit removal. At 60 and 80^{o}C, deposits were totally removed in all cases. Increasing the chemical concentration reduced cleaning time of egg yolk deposit and sodium hydroxide solution gave shorter cleaning time than LAS solution. Increase in hydration temperature, hydration time and pre-soaking chemical concentration result in lower adhesive and cohesive strength of egg yolk deposits. The adhesive strength was found to be greater than the cohesive strength in all cases. It can be concluded that temperature dominates the swelling and cleaning of egg yolk deposits while mechanical and chemical action also play an important role in swelling and cleaning of egg yolk deposits

ACKNOWLEDGEMENTS

Bismillahirrahmanirrahim, I would like to express my gratitude to those who have assisted me to complete this thesis. First of all, Alhamdulillah, Allah has given me this big opportunity to complete my study and blesses me with the courage and strength. My special thank goes to my beloved parents, siblings, and all family members who always support me with full encouragement. Furthermore, I also would like to thank my supervisors, Professor Serafim Bakalis and Professor Peter fryer who are very helpful in providing me with a good supervision and guidance towards in the execution of experiments and writing of this thesis.

I am indebted to my friend Muhammad Hazwan Hamzah who is responsible to print out this thesis and submit it to the office because currently I am not at the university, and also to my sponsor MARA for the sponsorship throughout the years of my study.

Last but not least, I would like to express my sincere and heartiest thanks to Dr. Kylee Rebecca Goode and Dr Ourania Gouseti who will always be there for me and really helped me a lot during my data collecting and thesis writing period. Not to forget anybody who has helped me during the completion of my project and thesis, but was not mentioned here. I am very grateful for all your helps. Thank you. May Allah bless all of you, Amiin.

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LIST OF SYMBOLS AND ABBREVIATIONS

β-Lg	beta lactoglobulin
SCM	Sweet condensed milk
FDG	Fluid Dynamic Gauging
WPC	Whey protein concentrate
WPI	Whey protein isolate
δ	thickness (mm)
MHF	Microfoil heat flux sensor
NaOH	Sodium hydroxide
PHE	Plate heat exchanger
SEM	Scanning electron microscopy
CFD	computational fluid dynamics
w/v	weight per volume
U	Heat transfer coefficient
T_c	thermocouple

CHAPTER 1: INTRODUCTION

1.1 Research background

Cleaning of food fouling deposits has always been a major concern in the food processing industry. In part because cleaning requires resources to be spent on that plant that is not directly value added, especially if cleaning is not optimised. Not to mention the influence fouling may have on limiting product quality and safety (Changani *et al*., 1997). Hence, the cleaning process has been studied in detail and various models been proposed (for examples see: Fryer *et al*., 2006; Friis and Jensen 2002; Dürr and Graβhoff, 1999). The objective of cleaning research usually is to minimise the time involved by understanding the effect of flow velocity, chemical type and concentration, pH of the cleaning solution, the temperature and pressure on cleaning profiles and cleaning time (Huang and Goddard, 2015). A cleaning profile for protein based films has been determined from milk processing (Xin *et al*., 2002; Gillham *et al*., 1999; Bird and Bartlett 1995):

i) **Swelling -** alkali solution contacts the deposit and causes swelling, forming a protein matrix of high void fraction;

ii) **Erosion -** uniform removal of deposit by shear stress forces and diffusion; there may be a plateau region of constant cleaning rate, but this depends on the balance between swelling and removal.

iii) **Decay -** the swollen deposit is thin and no longer uniform; removal of isolated islands occurs by shear stress and mass transport.

The hydration (swelling) stage is one of the most crucial parts in cleaning of food fouling. During this phase the interaction between water and/or chemicals and the deposit takes place. Diffusion and reaction of the fouling with cleaning chemical will be important in the initial stages of cleaning as the deposit swells. If this phase is not optimised, cleaning times will be excessive.

It is important to study hydration kinetics of food fouling films in order to understand the mechanisms of swelling and fouling dissolution to ultimately minimise cleaning time. Swelling profiles for protein gels and protein based fouling deposits have been studied (for examples see: Gordon *et al*., 2010; Mercadé-Prieto *et al*., 2006; Tuladhar *et al*., 2002). Protein deposits are often removed in large chunks, and may remove minerals from different layers with them. Recent work has turned to studying fat based fouling (for examples see: Pérez-Mohedano *et al*., 2015 and 2016; Huang *et al*., 2012). The focus here is on thermally induced fat based deposit which is notoriously hard to clean, especially once solidified. In this research, hydration of a model food fouling deposit, egg yolk, is carried out under various conditions, to characterise swelling profiles and probable impact on cleaning times.

1.2 Development of fouling deposit

Food materials have the potential to stick to multiple surface types. For example:

(i) Soft, porous textiles used in everyday life. E.g. clothing, table and dishware

(ii) Hard surfaces (which may or may not be porous):

a. used at home *i.e. Metals (pans, cutlery), glass, plastics (containers and worktops), wood, ceramics (plates, bowls), enamel (teeth, coatings for pans/crockery).*
b. used in industry *i.e. metals (pipework, tanks, heat exchangers etc…), glass, plastics (gaskets).*

Food fouling may also form under different conditions as a result of thermal treatment (*e.g. in cooking pans or pots in the home/heat exchangers in the plant*) or may be non-thermally induced (e.*g. food spillages and dehydration on clothing and crockery*). The thermal treatment of foods is a typical food processing operation used to pasteurise, concentrate and dry products. Foods undergo structural and chemical changes during the heat treatment and as a result some constituents adsorb to the heat transfer surface resulting in fouling. During food fouling, both organic and mineral deposits can be formed, as is the case with milk fouling, by a combination of precipitation and crystallisation (Changani *et al*., 1997). Various authors have tried to model fouling phenomena for heat induced deposits (for examples see: Yang et al., 2012; Grijspeerdt *et al*., 2004; de Jong *et al*., 2002, Schreier and Fryer 1995). The type of deposit and formation mechanism, depend on the food components present and the processing conditions used. Altering composition can have significant influence on both the fouling and cleaning profiles observed (Huang and Goddard 2015; Christian *et al*., 2002).

1.3 Thesis objectives

The aims of the thesis were;

(i) To study the effect of various parameters on swelling and removal of egg yolk deposits during hydration.

(ii) To investigate the effect of chemical and mechanical action on swelling and removal of egg yolk deposits during hydration

(iii) To study the effect of various parameters on cleaning of egg yolk deposits at laboratory scale and the adhesive and cohesive strength of egg yolk deposits.

1.4 Thesis outline

This thesis consists of 7 chapters including this chapter as the introduction to the thesis where the research background, the development of fouling, the thesis aims and the thesis outline were discussed.

Chapter 2 discusses the review on cleaning studies which includes the fundamental of the materials used in this study, the development of method and technique used in this study, and published works from other researchers on cleaning.

Chapter 3 describes materials and methods used in this study including preparation of sample, experimental equipment set-up, experimental procedures and protocols, and data analysis methods.

Chapter 4 discusses the effect of different temperature, cleaning fluids, stirring speeds, chemical concentrations, and sample configurations on swelling and removal of egg yolk deposits during hydration. Results obtained from the experiment carried out were presented and discussed in this chapter.

Chapter 5 focuses on the effect of chemical and mechanical action on swelling and removal of egg yolk deposits during hydration at room temperature. All findings from the experiments conducted were presented and discussed in this chapter.

Chapter 6 concentrates on the effect of various experimental parameters i.e temperatures, chemical concentrations, and different cleaning chemicals on cleaning of egg yolk deposits using cleaning rig (previously known as flow cell rig or coupon rig). This chapter also focuses on the effect of different pre-soaking temperature, time, cleaning chemicals, and chemical concentrations on the adhesive and cohesive strength of egg yolk deposits. Results obtained from the experiments conducted were presented and discussed in this chapter.

Chapter 7 discusses on the general conclusions of all results and findings from all experiments throughout the study conducted and some future work recommendations which are potentially to be carried out.

CHAPTER 2: REVIEW OF FOULING AND CLEANING STUDIES

2.1 Review on materials

2.1.1 Egg yolk chemical composition

Egg yolk is known to have lower protein content and higher fat content compared to egg white with a protein content of 16% and fat content of 34% (Burley and Vadehra, 1989; Acker and Ternes, 1998; Guilmineau, 2008). The total dry matter of freshly laid yolk is about 52 % + 0.2% of its composition and increases slightly as the laying hen get older for an average of 2 % (Guilmineau, 2008). Guilmineau (2008) also reported that, during storage of the eggs in the shells, the transfer of water from the white into the yolk resulted in a range of variety in egg yolk dry matter. The different osmotic pressure in the white (250mOsm) and the yolk (320mOsm) has also caused the water to transfer from the white into the yolk. Staldermann and Cotterill (1986) notes that increase in pH of the egg yolk during storage will change the physical properties of the egg yolk and thus allows a slow diffusion of water and small molecules from the white to into the yolk. Table 1 shows the general average composition of pure fresh egg yolk (Burley and Vadehra, 1989; Acker and Ternes, 1998; Guilmineau, 2008). All lipids in egg yolk are found to be associated in lipoprotein complexes. Lipids comprise of 62 % triglyceride, 33 % phospholipids, and less than 5 % cholesterol. Carotenoids, which give the yellow/orange colour to the yolk, represent less than 1 % of total lipids.

Table 2.1 Detailed average composition of fresh egg yolk

Component (% w/w)	Subgroup	Main molecule	Content
Water 48			
Lipid (34 %)	Triglyceride	FA (C 16:0), PUFA (C18:2) 22.6	
		MUFA (C18:1)	
	Phospholipid (9.6 %)	Phosphatidylcholine (=lechitin) 7.0	
		Phosphatidylethanolamine 1.4	
		Sphingomyelin	0.6
		Lysophosphatidylecholine	0.2
		Lysophosphatidylethanolamine	0.2
		Plasmogen	0.1
		Inositol phospholipid 0.01	
		Sterols	Mainly cholesterol 1.8
		Proteins (16%)	Phosvitin 1.8
		Lyvetins (α-, β- γ-)	5.0
		Lipovitellin (α-, β-) = HDL-apo	5.8
		Lipovitellinin =LDL-apo	3.5
Carbohydrates		Free glucose	0.2
Vitamins		Vitamin A, D, E, K, B1, B2, B6, B13	0.8
Minerals			P, Ca, N, K, Cl, S, Mg, Fe 1.0

FA: Fatty acid, PUFA: Poly unsaturated fatty acid, MUFA: Mono unsaturated fatty acid.

Based on data from: Burley and Vadehra (1989); Acker and Ternes (1998); Guilmineau (2008)

Egg yolk can also be defined as an emulsion of lipid droplets dispersed in an aqueous protein solution (Strixner and Kulozik, 2011) comprising three parts known as spheres, granules and profiles (LDL). Bellairs (1961) described that spheres representing only about 1 % of the dry matter of egg yolk, which made of lipoproteins, have 4 to 15 μm diameter and appear as droplet aggregates in microscopy, while Yang (1987) has reported that the experimental results show that the spheres structure can be removed by a simple manual agitation and this bring to a conclusion that they only present in the unbroken egg. Previous studies by Bellairs (1961), Burley and Vadehra (1979), and Chang et al. (1977) indicates that the granules part have the average of 0.8 to 2 μm diameter, 1.089 g/ml and 1.210 g/ml density, and have a spherical and more or less flatten shape. The granula particles can be found with a diameter up to 10 μm. There have been reported that the profiles can be defined as a low density lipoprotein which has a range of diameter between 12 to 60 nm (Chang *et al*., 1977; Martin *et al*., 1964). Huopalahti *et al*. (2007), and stated that LDL particles are also found in the profiles and Martin *et al*, 1964 stated that these structures are very low density lipoprotein or merged LDL which are similar to what have been reported by Ala-Korpela *et al*. (1998) for human plasma LDL. Strixner and Kulozik (2011) described that the orange translucent liquid plasma represent 52 to 58 % of all proteins, 85 % of phospholipids of egg yolk and 75 to 81 % of egg yolk dry matter. The lipid/protein ratio in granules was reported to be around o.5 whereas for the egg yolk, it was reported to be around 1.9 and this bring to a conclusion that granules are more protein-rich than plasma and egg yolk (Dyer-Hurdon and Nnanna, 1993). 42 to 48 % of the proteins and 15 % of the phospholipids in egg yolk were reported to be found in granules (Burley and cook, 1961; Causeret *et al*.,

1991; Anton and Gandemer, 1997) and these make them to represent 19 to 25 % of the total dry matter of the egg yolk. Despite reported to contain over 98 % of the iron, 81 % of the calcium and 70 % of the magnesium of the egg yolk, granules also contain most of the di- and tri-cations (Causeret *et al*., 1991), while plasma was described by Backermann (2007), and Causeret *et al*. (1991) to contain over 90 % of the potassium and 92 % of the sodium of the egg yolk, and most of the monovalent cations.

Studies by Le Denmat *et al*. (2010) and Assis *et al*. (2010) found that egg yolk proteins have different solubility in different pH and temperature regions. The proteins in egg yolk have an average isoelectric point of 5.3 and in this condition protein has lower solubility (Gaonkar *et al*., 2010). In terms of emulsifying properties, egg yolk has played a major role in food emulsion products such as mayonnaise, dressings, sauces, custards, puddings and ice creams (American Egg Board, 2013). Egg yolk helps to stabilize emulsions by creating an interfacial film to prevent two immiscible liquids from separating. The composition and the concentration of this interfacial film affect the physico-chemical properties emulsions (Anton & Gandemer, 1999; American Egg Board, 2013; Soderberg, 2013). It is known that the protein components in egg yolk are the key players in egg yolk emulsification (Soderberg, 2013). Anton & Gandemer (1997) earlier suggest that these protein components are lipoproteins (LDL and HDL), phospholipids, and the proteins phosvitins and livetins, where low density lipoproteins (LDL) are the most crucial ones for food emulsions. Kurt and Zorba (2009) have carried out a study on the effect of different pH on food emulsifications. They found that pH influences the egg yolk proteins emulsifying properties which showed that the emulsion capacity increased with an increase in pH and the optimum values for emulsification

were between 4.61 and 7.43. For example, mayonnaises are made at pH 4 while béarnaise are made at pH 7 (Anton & Gandemer, 1999). Egg yolk also is said to be a flexible emulsifier as it can stabilize emulsions in cold (mayonnaise) and warm (hollandaise sauce) condition (American Egg Board, 2013; Soderberg, 2013).

Egg proteins are the most effective proteins to form foams. These foaming properties are important in certain baked and confectionary products (Yang and Baldwin, 1995). The mixture of different proteins gives egg white good foaming properties (Macdonell, 1955; Stadelman and Cotterill, 1995). Lomakina & Mikova (2006) suggest that the foaming properties off egg white are affected by environmental factors such as temperature, pH and ionic strength. They also found that egg white shows better foaming in room temperature and under this condition, the foam formation is quicker and the foam volume is greater (Soderberg, 2013). However, earlier research by Kim & Setser (1982) indicates that egg yolk was found to decrease the foaming capability of white egg, and it is almost impossible to separate egg white completely from egg yolk. Soufflés and omelets are the foam products that contain both egg white and egg yolk. The whole egg, or just the egg yolk alone, can also be used to make sponge cakes (Yang and Baldwin, 1995; Soderberg, 2013). Another highly important egg protein functional property in food industry is gelation or coagulation properties (Campbell *et al*., 2003). The eggs ability to form gel networks upon heating plays a crucial role in the making and texturization of foods like cakes, creams, omelets, confectionaries and sauces (Kiosseogglou, 2003). The egg protein molecules will aggregate and form insoluble networks (a gel or coagulum) in baking process, and it is these networks that give cakes and muffins their heights volume and stability (American Egg Board, 2013;

Soderberg, 2013). Apart from that, it is known that egg yolk proteins also form gels. Kiosseogglou

(2003) suggests that this gel formation property is a function of the apolipoproteins in the low-density lipoprotein micelles.

2.1.2 Stainless steel used in food industry

In food industry, stainless steel is used as a material to fabricate most of the processing equipment because of hygienic reason (Holah & Thorpe, 1990). It has a sufficient level of corrosion and oxidation resistance for food processing industry (Santos *et al*., 2004). This corrosion resistance comes from an invisible protective oxide films known as “the passive layer” which built up on the stainless steel surface spontaneously under the influence of oxygen from air and water. This passive layer or film is known to consist of chromium oxide and hydroxide (DeBold, 1988; Banes, 1990; Storer, 1997) plays a crucial role in corrosion resistance affects molecular adsorption properties (Mantel *et al*., 1995). If this film was physically or chemically damaged, it will rapidly repair itself once the source of damage is removed, and the surface is exposed to oxygen again (Davis *et al*., 1994; Tuthill & Corvett, 2000). Apart from that, it is easy to create smooth and non-absorbance surface on stainless steel. Stainless steels are also tolerant to the wide range of temperatures used in the food processing industry and have thermal shock resistance ability (Faille *et al*., 2000). The physical properties of stainless steel make it suitable for food processing industry because they are strong, stable, can be formed into any desired shape and easy to be cleaned or maintained without dismantle it (Cleaning in place) (Tuthill *et al*., 1997). Austenitic stainless steels are commonly used

in food processing equipment (Sundgren *et al*., 1985; Hayashi *et al*., 1989; Davis *et al*., 1994) because of their price and have relatively good corrosion resistance in the contact fluid as compare to other types of stainless steel (Nagai, 1995; Suzuki *et al*., 1998). Fukuzaki *et al*. (1997) have carried out a research to study the isoelectric point of particles of type 316 austenitic stainless steel suggested that adsorption of electrolyte molecules is a function of the density of the hydroxyl group of the stainless steel. The hydroxyl group of the stainless steel surface may dissociate depending on pH values. This surface hydroxyl group plays an important role in adsorptions of molecules, particularly in the case of molecules having dissociating groups, such carboxyl group (Fukuzaki *et al*., 1995; Fukuzaki *et al*., 1998; Hubbard, 2002). Stainless steel equipment surfaces may adsorb various manufacturing process fluid ingredients. A lot of researches have been carried out to improve cleaning techniques for removing fouling deposits from the contact surfaces of stainless steel and prevent or minimize fouling on the equipment stainless steel surfaces (Krysinski *et al*., 1992; Grant *et al*., 1996; fukuzaki *et al*., 1998) because most manufacturing process such as those in pharmaceutical and food processing plants require unfouled clean surfaces of the manufacturing equipment for maintaining sanitary conditions, assuring quality of the products, and attaining equipment efficacy (Sandu & Lund, 1985; Villafrance and Zambrano, 1985; Itoh *et al*., 1995; Hubbard, 2002). Stainless steel was used in this study as a fouling surface.

2.1.3 Surfactant and detergent

Surfactant can be defined as a surface active agent (Jonsson *et al*., 1998), which acting to reduce the surface tension of the medium in which it dissolved. Surfactant presents in 15 to 40 % of the total detergent ingredient which make them the most crucial ingredient in household and laundry leaning products (Scheibel *et al*., 2004). It can be divided into four types of groups which is anionics, nonionics, cationics, and zwitterionics (Bing Shao, 2011). Anionic surfactants are a type of surfactants that are widely used because of its low manufacturing cost. Soap, which is made from animal fats or vegetable oil that is allowed to react with an alkali, is the first anionic surfactant to be invented (Scheibel *et al*., 2004). However, it was found that soap is sensitive to hard water and does not work well in lower temperature and cold water. These limitations have been a major driving force for the development of synthetic anionic surfactants (Yu *et al*., 2008). Alkyl benzene sulfonates (ABS) were the first synthetic anionic surfactant to be introduced and used in the detergent compositions. However, this ABS could not be broken down by microbes, and thus they left foam in river water. Then, biodegradable surfactants known as linear alkyl benzene sulfonates (LAS) were invented to replace the ABS. Jonsson *et al*., 1998; Scheibel *et al*., 2004). It is widely understood that an LAS surfactant will be sequestered and be precipitated by divalent cations under high water hardness condition which reduces the detergent cleaning power. In order to overcome this situation, low levels of alkyl ether sulfates (AES) have been used in a surfactant system (Prada-Silvy *et al*., 2000). Alpha olefin sulfonates (one of the anionic surfactants) which play a similar function to AES have been widely used because of its superior performance characteristics and enhance biodegradability (Scheibel *et al*.,

2003; Yu *et al*., 2008). There are several researches have been carried out to study and understand this phenomenon, Sanderson *et al*. (2006) have conducted a study to investigate the risks caused by alkyl sulfates, alkyl ether sulfates and linear alkyl benzene sulfonates in river water and sediments, and found that they showed low aquatic risks.

Detergent usually contains a mixture of different type of surfactants to make sure that it does not affect hands skin, and increase their cleaning performance (Motson, 1999). Surfactant molecules identically have a hydrophilic head molecule which is attracted to water molecule and hydrophobic tale that repels water molecule and thus simultaneously attaches itself to soil particles. Both of them play a key role in cleaning by reducing the interfacial surface tension between two media or interfaces (water/air, water/soil, and soil/surface). This lower surface tension of the water makes it easier to lift the soil off the surface and help to keep them suspended in soiled water. The hydrophilic head of the surfactant molecules will remain in the water and pull the soil towards the water, away from the surface. They then suspend the soil particles in water to remove them. (Jonsson *et al*., 1998). Romaninho et al. (2006) had used an alkaline detergent which was a mixture of non-ionic and anionic surfactants at 2.0% w/v to study the effect of different stainless steel surface material on milk components fouling.

In this present study, a type of LAS solution was used as a surfactant. LAS (linear alkyl benzene sulfonate) are a kind of anionic surfactant which is environmental friendly due to its biogradable property. LAS also work effectively at lower temperature water unlike the fatty acid derived soap (Yu, *et al.,* 2008). A lot of researches on LAS have been

done because it plays an important role in cleaning industry for over the past 70 years (Scheibel, 2004).

2.1.4 Sodium hydroxide in cleaning

There are 3 types of forces that involved in cleaning which are mechanical force, chemical force and thermal force. This section will discuss about chemical force involved during cleaning process, particularly the use of sodium hydroxide. Sodium hydroxide is known to be a common and widely used chemical in cleaning. Sodium hydroxide acts as a cleaning agent by saponifying fats and dissolved proteins (Block, 1991). It also can solubilize precipitated proteins and enhance its hydrolyzing power in the presence of chlorine (Jungbauer *et al*., 1994). Alkaline detergents are usually used because of their ability to dissolve protein, fat, and sugars. It can be a pure sodium hydroxide or sodium hydroxide based formulated detergent. In most cleaning applications, sodium hydroxide is usually used at 0.5 to 2 wt% concentration. A study by Bird and Bartlett (1995) reported that the optimum cleaning concentration in terms of cleaning time for whole milk deposit was at concentration 0.5 wt%. Milk fouling contains large degree of whey proteins that have denatured and aggregated through various crosslinking reactions. Higher concentration of sodium hydroxide will induce crosslinking between them and make it harder to remove, thus increase the cleaning time. Christian and Fryer (2003) used 0.1, 0.5 and 1 wt% of sodium hydroxide as cleaning agent for their study. In this research they found that chemical removal dominates above 0.5 wt% of NaoH at temperature of 50^{o}C and above for all flow rate, and physical process dominates when the chemical (NaOH) is low. They also stated that the strength of the deposit is affected by chemical, and foulant removal after swelling is a function of

chemical force as the cleaning agent dissolves the entire deposit, and little or no isolated islands removal is observed. Liu *et al*. (2003) had also used sodium hydroxide solution at 0.5wt% concentration to study the effect of cleaning chemical on the removal of milk protein deposits. It was found that the whole deposit could be removed after the sample was soaked in 0.5 wt. % of sodium hydroxide solution as compare to soaking in water which shows that it was difficult to remove all the deposit from the surface when no chemicals were used. The similar concentration of sodium hydroxide solution (0.5 wt %) was also used by other researchers. Rosmaninho & Melo (2006) used 0.5 wt% of sodium hydroxide in their study to investigate the effect of different properties of stainless steel surface on milk components fouling while Chen and Ozkan (2002) has used the same concentration of sodium hydroxide in their research in order to study the cleaning rate of whey protein gel deposits in the uniform cleaning stage. In this present research, Sodium hydroxide was used as cleaning agents besides LAS solution and commercial detergent solution (Arial).

2.2 Review on fouling behaviour and properties

2.2.1 Effect of surface adhesion

The development of fouling is a multiple stage process where adhesion of the fouling agents to surface is a necessary step (*Oliveira*, 1997). Adhesion is the tendency of different materials or surfaces to cling to each other. Fouling happens when gravitational forces are not significant. This means that the particulate materials are not large enough to be removed by the gravitational or hydrodynamic forces from the surface (*Oliveira*, 1997). It is known that *van der Waals* forces are the main attractive forces between the particulate materials. However, electrostatic double-layer forces can

be developed between them if the particles are immersed in liquid medium. Other types of interaction such as hydrophobic interactions in polar media, ion bridging and steric interactions in the presence of polymers can also play major roles in fouling adhesion.

2.2.2 Fouling mechanisms

Fouling can be defined as the development of unwanted material build-up on a surface (Goode et al., 2013). Fouling deposits formed as a result of adhesion of species to the other species and cohesion between elements of the material (Liu et al., 2006). It can be formed from carbohydrate, protein, lipid, and mineral residues. It has been suggested that denatured protein deposits are the most difficult to clean due to the type of chemical bonding within deposit (Aziz, 2008). In the case of milk fouling, fouling starts as soon as a dairy product is brought into contact with a stainless steel surface albeit at a molecular level and invisible to the naked eye. It is believed that it starts with whey protein adhesion at room temperature. Upon heating, the whey proteins start to unfold and expose a free S–OH group, changing into an activated state which allows attachment to the protein layer initially formed (Visser & Jeurnink, 1997; Rosmaninho *et al*., 2005). This first protein layer, however, is affected by the deposition surface since protein adsorption onto solid surfaces is the result of several interactions occurring between the protein, the surface and the solvent with other solutes present in the system (Haynes & Norde, 1994; Santos *et al*., 2005). These interactions are significantly influenced by the surface properties, more precisely its surface energy. Another major contribution to fouling in milk processing is the presence of calcium and phosphate ions. Upon heating of milk, part of these ions will tend to precipitate as a

calcium phosphate salt (owing to the inverse solubility of calcium phosphate with temperature) which ultimately will form a mineral deposit on the stainless steel surface in a typical crystallization fouling process (Bott, 1995; Krause, 1993). Even when the deposits were predominantly proteinaceous, a calcium phosphate layer was found next to the surface due to migration of mineral ions (Changani et al., 1997).

Previous studies found that the amount of fouling could be reduced by preholding milk at 75^{o}C for 10 minutes, thus suggested that fouling was due to the denaturation of protein and decrease in solubility of milk salts with increasing temperature (Bell and Sanders, 1944; Changani et al., 1997). Christian and Fryer (2003) proposed that the chemical composition of the deposit is different to composition of the fluid. Burton (1988) has named two types of deposit that formed when milk undergoes heat treatment as Type A and Type B. Type A is high in protein content and forms at temperatures of 85 ^{o}C to 110^{o}C due to the denaturation aggregation of proteins (mainly β-lactoglobulin) while Type B deposit forms at temperatures of 110 to 140 is mainly consists of calcium and phosphorus which form inversely soluble salts (Christian & Fryer, 2003). Changani et al. (1997) proposed that dairy fouling is depending on several physicochemical factors such as protein denaturation, protein aggregation reactions, insolubilization of calcium phosphate and change in pH solution during heating. Several previous studies have been carried out in order to investigate the factors that affect milk fouling in food processing industry, with Parris et al. (1998) reported that generic variants of β-lactoglobulin exhibited different degrees of fouling while Burton (1998) suggested that cattle feeding regimes affected deposition although Grandison (1998) reported no correlation between diet and Fouling. Fryer et al. (1998)

found that there is a positive linear correlation between the total amount of deposit from whey protein concentrate and protein concentration. Jeurnink (1999) also reported that fouling and calcium deposition increased with serum protein concentration. Schraml and Kessler suggested that fouling increased to a maximum concentration of 25% total solids after which deposition decreased. Rosmaninho et al. (2006) had carried out a research to investigate the effect of different stainless steel surface properties on milk components fouling. It was found that the fouling behaviour was influenced by the surface material, although in different ways for the deposition or the cleaning phases.

There are also several previous studies on food fat fouling. Huang *et al*. (2010) studied food fat freezing fouling using spinning disk apparatus (SDA) and CFD simulation to study the heat transfer, surface temperature and shear stress, while Huang *et al* (2011) studied the effect of deposition time, disk rotational speed of SDA, and temperature driving force on tripalmitin (PPP) freeze fouling behaviour which reported that deposition appears to be driven by gel formation at the shear and temperature conditions acting at the surface. Nigo *et al*. (2009) reported that a novel fouling apparatus SDA has been developed to study fat freezing fouling behaviour using model solutions of PPP in paraffin. This study found that fat fouling can be monitored in situ and the laminar flows in the SDA can be simulated using CFD techniques as well as highlighted the importance of gel formation conditions on fouling behaviour. Huang *et al*. (2013) carried out a set of experiments to investigate the influence of starting mode on the formation of fouling deposits using modified spinning disk apparatus and they found that surface temperature is the key factor in this fouling formation, initial fouling behaviour is very sensitive to the start-up protocol, the constant and falling rate fouling regime parameters

were not affected by the start-up mode, deposit exhibited significant ageing, increasing in solids content over time and low fat concentration gave weaker gels which could be removed by shear. In this present study, egg yolk powder is used to produce fouling deposit because egg yolk contains more than 40% proteins and lipids, and it is easier to obtain reproducible fouling behaviour from egg yolk powder compares to egg yolk from chicken eggs.

2.2.3 Cleaning map and classification of cleaning problems

Fryer and Asteriadou stated that there are two factors dominated the cleaning process; type of deposit, and type of cleaning detergent. A cleaning map was proposed in Figure 1.2 to demonstrate fouling and cleaning problems investigated. It was a two-dimensional coordinate system, the axes of which represented type of deposit and type of cleaning detergent respectively. Each point on the coordinate embodied the correspondence between deposits and cleaning fluids (Fryer & Asteriadou, 2009). Many practical problems could be found in the map, including rinsing with water to clean stains on packaging (non-viscous fluids, hot water) and cleaning during changeovers of different products to prevent cross-contamination (viscous fluids, water at ambient).

Three types of deposits which were the most difficult to clean were defined, presented in the shadow area (Fryer & Asteriadou, 2009). Type 1 represented the viscous fluid foulants which could be removed by water action alone, including toothpaste and shampoo. Type 2 represented the organism foulants in which biocide must be brought in to kill biofilms. Type 3 represented the cohesive solid foulants which could not be removed by water alone, which was the most common practical issue in chemical

industry. Cleaning detergent must be introduced either to dissolve the foulants or to alter the foulants into a cleanable form by chemical reaction. Acidic and alkaline solutions were normal practical choices in industry, depending on the characteristics of the deposits (for example acidic for minerals and alkaline for proteins).

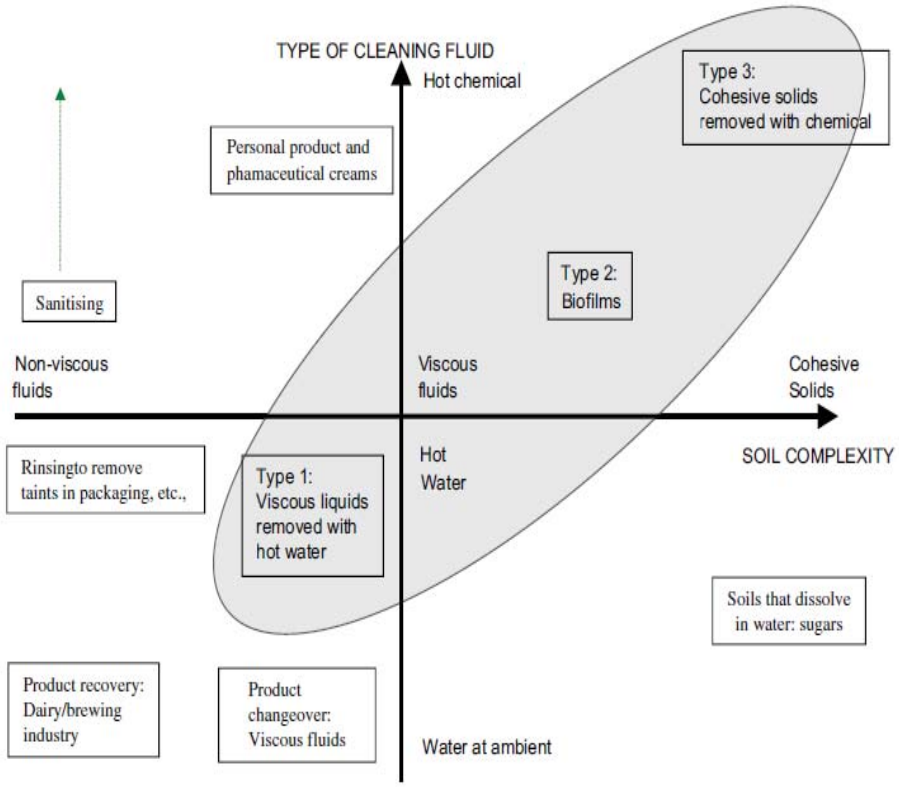


Figure 2.1: Cleaning map and three types of deposit (Fryer & Asteriadou, 2009)

2.2.4 Cleaning of fouling deposit

Cleaning is a multistage process consisted of various steps that may be controlled by chemical reaction, mechanical action and mass transfer (Dong Chen & Ozkan, 2004). The cleaning process can be described as a three-stage process based on the cleaning periods; i) deposit swelling on contact with chemical, ii) removal of “uniform” swollen layer by shear/ mass transport, iii) removal of isolated “islands” by shear/mass transport

i.e decay stage (Gillham et al., 1999). Grassoff (1997) has described the cleaning process where initially components of cleaning solution are transferred to the foulant surface, then the "*decay* and *swelling*" reaction takes place where the diffusion of cleaning agents into the deposits occurred which swells the deposit rapidly. This diffusion process would not complete for the whole deposit and the top layer could be removed because of the deposit has been weaken by partial hydrolysis and insertion of water molecules. Then, the cleaning fluid will continue to diffuse on the remaining deposit until the metal surface is reached and at this point, the deposit becomes totally swelled. At the *erosion* stage, the remaining deposit attached to the surface will be removed by cleaning solution. The size of deposit aggregates removed is depending on the flow rate of the cleaning solution and the inner structure of the deposit. Bird (1992) found that only about 5% of the swelling deposit remains on the surface after 3 to 10 minutes of cleaning. If the wall shear stress exceeds the normal adhesion force, then fluid shear is needed to transport the weak part away from the deposit body. At the third stage, any mineral layer that presents as a thin sublayer will be removed if proper cleaning agent is used (Grassoff, 1997).

Some of the previous researches have indicated that the dissolution of deposits in the cleaning solution is one of the key players that control cleaning process especially in the plateau or uniform stage (Bird & Fryer, 1991; Gillham et al., 1999; Xin et al., 2002) and Bird and fryer (1991) and *Gallot-Lavallee* and *Lalande* (1984) also suggested that the cleaning process is a function of hydroxide transport and surface removal steps. An empirical model based on transportation of reaction products through a boundary layer has already been developed *(Schlussler*, 1970) in order to explain the removal porous

deposits from a heat transfer surface. *Gallot-Lavallee & Lalande* (1984) has also reported that in the case of milk fouling, the measured cleaning rate was proportional to the concentration gradient of the intermediate reaction products and the mass transfer coefficient of the reaction products at the interface. In this research, the interaction between the cleaning solutions and the deposit will be studied in order to gain knowledge about the cleaning process.

2.2.5 Cleaning mechanism of type-3 deposits

In order to achieve cleaning, two forces must be overcome (Figure 2.7); the interior cohesive forces which binding the foulant together, for example Van der Waals force and electrostatic forces, and the adhesive forces between the foulant and the surface, which was proportional to the contact area (Fryer & Asteriadou, 2009; Goode, 2012). A minimum free energy within the deposit-surface system was found to enable fouling according to DVLO theory (Goode, 2012):

$$\sqrt{\gamma_S} = \frac{1}{2}\left(\sqrt{\gamma_F} + \sqrt{\gamma_C}\right) \qquad (1)$$

Where γ_S, γ_D and γ_F represented the Lifshitz-VDW free energy of the surface, foulant and cleaning stream respectively, which were measurable by the contact angle method (Zhao et al., 2004). It was found that the removal energy was 20-25 mN/m for the fouling of baked and unbaked tomato soil on 316 L stainless steel (Liu et al., 2006).

For the cleaning of type 3 deposits, cleaning chemical must be introduced either to dissolve the foulants or to change the foulants into a cleanable form by chemical reaction, which was categorized as diffusion-reaction removal (Fryer & Asteriadou, 2009). The cleaning effect depends on the mass transfer of cleaning agents from fluid to surface, the diffusion of active components within the deposit and the rate and effect of

chemical reaction.

Most type 3 deposits were hydrophobic protein-type foulants which swollen once contacting with alkali, and a high-void structure was formed to make it easier to be removed by shear stress. It was proven that protein deposits could easily be cleaned off after soaking in alkaline solutions, even with water alone (without chemical) (*Christian & Fryer*, 2006). *Goode* (2012) has characterized a clear three-stage procedure for protein cleaning; swelling, where a cleanable form is created by chemical reaction; erosion, where the cohesive forces inside the deposit failed and the deposit is removed evenly, which is controlled by diffusion and shear stress; decay, where the adhesive forces between the deposit and the surface failed and the deposit is removed by chunk, which is controlled by mass transfer and shear stress.

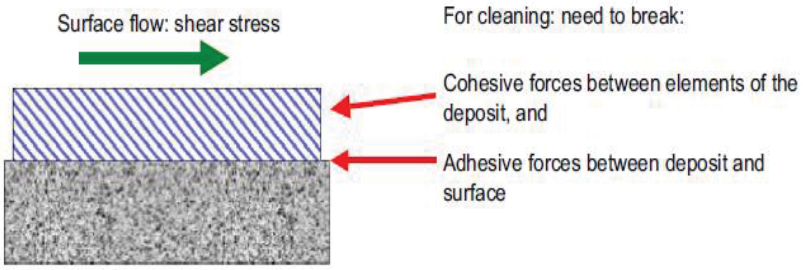


Figure 2.2: Forces need to be overcome for cleaning (Fryer & Asteriadou, 2009)

2.3 Review on hydration and swelling fouling deposit

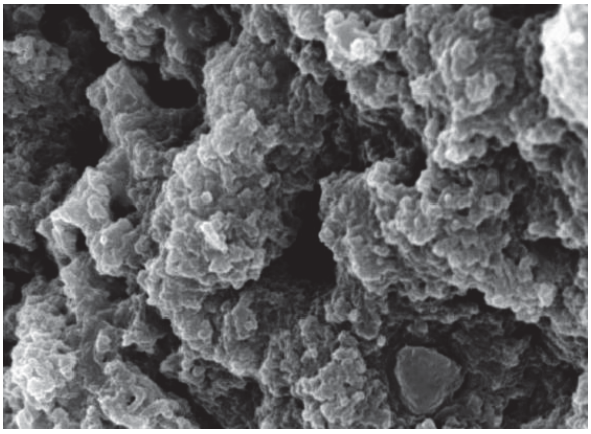
2.3.1 Soaking and hydration studies

It is well understood that hydration, swelling and cleaning of deposit are related. Moisture content plays an important role in physical and chemical properties of food samples (Labuza and Hyman, 1998). Perez-Mohedano et al (2016) reported that

absoprtion of water or liquid into a food sample may happen when the food sample which low hydrated are contacted with high moisture or liquid environments. Thus it would lead to an increase in the volume of the food sample i.e. swelling. Perez-Mohedano (2016) stated that when the thermodynamic equilibrium within the control volume changed, swelling would happen. It is also understood that swelling of deposit would ease the deposit cleaning hence reduce the cleaning time (Grassoff, 1997)

Liu et al. (2006) studied the effect of pre-soaking treatment on the strength of the whey protein concentration deposit (WPC)(figure 2.3). Swelling phase occurred when the WPC deposit were pre-soaked with 0.1, 0.5 and 1 % sodium hydroxide solution for 60 minutes at 20^{o}C while no swelling observed for sample soaked in water at similar conditions. The deposit thickness was found to reach a maximum (plateau) after being submerged for a certain time. Increasing the caustic solution concentration changed the time taken to reach the maximum thickness and increased the maximum thickness of the WPC deposit. At 0.1 % NaOH, the time taken to reach maximum thickness is 5 minutes and the maximum thickness of the deposit was increased by 9 % compares to samples submerged in water. At 0.5 % NaOH, it took 25 minutes for the deposit to reach the plateau and the maximum thickness of deposit was raised by 53 % compares to samples soaked in water. At 1 % NaOH, the deposit took 10 minutes to reach the maximum thickness and the maximum thickness of deposit was increased by 100 % compares to samples submerged in water.

a)



b)

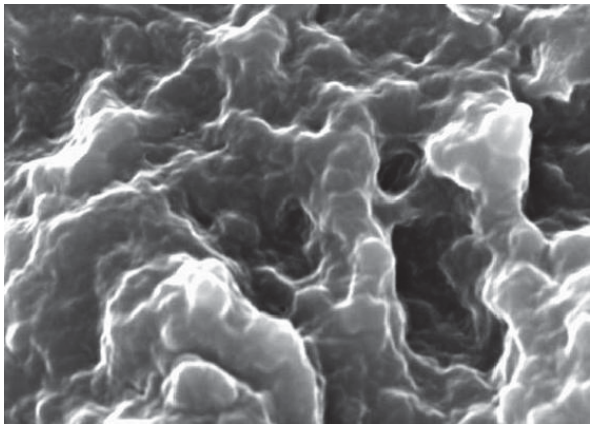


Figure 2.3: SEM pictures of native WPC deposits (a) and that after submerging in 0.5% NaOH solution for 5 min (b) at 20^{o}C (Liu et al., 2006)

Liu et al (2007) studied the effect of pre-soaking treatment on the strength of the egg albumin deposit (figure 2.4). Swelling phase occurred when the egg albumin samples were pre-soaked with 0.1, 0.5 and 1 % sodium hydroxide solution for 60 minutes at 20^{o}C while no swelling was observed for sample soaked in water at similar conditions. Increase in caustic solution concentration leads to increase in the maximum thickness reached and the thickness increament rate of egg albumin deposit. It was also found

that increase in sodium hydroxide concentration helps to increase the diffusion of chemical into the deposit and increase the extent of reaction of the protein.

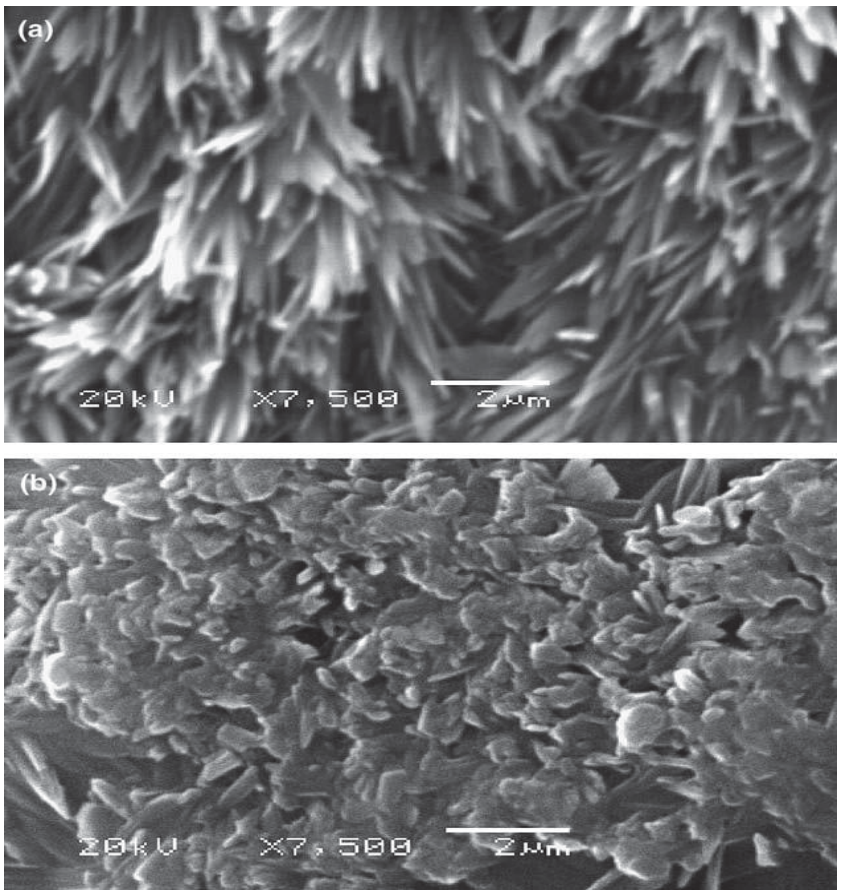


Figure 2.4 SEM pictures of (a) native ovalbumin deposits (b) and after submerging in 0.5% NaOH solution for 60 min at 20^{O}C by Liu et al. (2007)

Hagiwara (2015) studied the effect of acidic chemical pre-treatment on protein adhesion of stainless steel surface with egg white protein was used as the fouling material and a mixture of citric and nitric acid was used as the pre-treatment chemical. The following equation was used to determine the protein adhesion on stainless steel surface;

$$q_A = \frac{\Delta C \times V}{W_{ss} \times S} \qquad (1)$$

The effect of different temperatures on the egg white protein adhesion was studied. Figures 2.5 shows that samples with pre-treatment have lower protein adhesion compares to those samples without treatment. However, it was found that increasing adhesion temperature increased the protein adhesion of samples with acid pre-treatment while adhesion temperature shows only a slight effect on the protein adhesion of samples without treatment. Figure 2.6 shows the reduction ratio of protein adhesion with acid pre-treatment. It is shown that increase in adhesion temperature results in decrease of reduction ration of protein adhesion. Thus, it can be concluded that acid pre-treatment helps to suppress the protein deposit formation and enhance the cleaning of protein deposit.

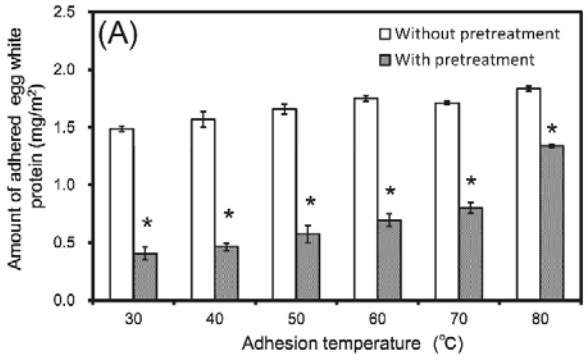


Figure 2.5: Citric acid as a pre-treatment technique for stainless steel surface (Hagiwara et al., 2015)

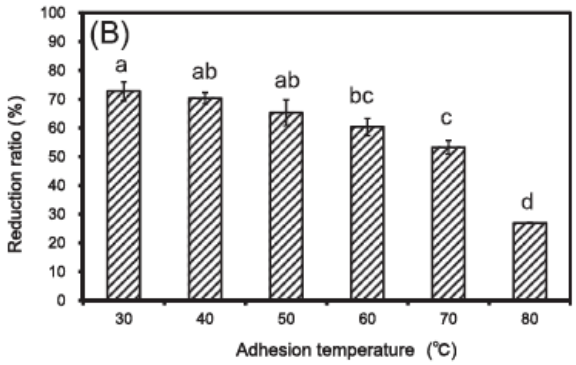


Figure 2.6: Reduction ratio by pre-treatment results (Hagiwara et al., 2015)

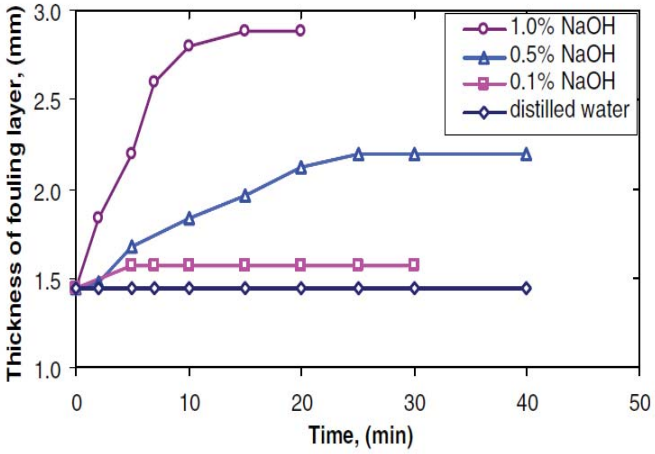
Anton and Gandemer (1997) reported that the solubility of egg yolk depends on its ionic strength. The authors found a low solubility (10 % solubility) of egg yolk samples were observed at low concentration of sodium chloride. Increasing the sodium chloride concentration from 0.1 to 0.3 M increased the solubility of egg yolk samples by 80%. However, increasing the sodium chloride concentration to 5 M did not increase the soulibility of egg yolk samples. It seemed like the egg yolk solubility has reached the pleateau at 3 M concentration of sodium chloride or more.

In this present research, the hydration of egg yolk deposit fouled on stainless steel surface will be studied in terms of moisture content, swelling performance, and the removal of deposit during hydration of egg yolk deposit.

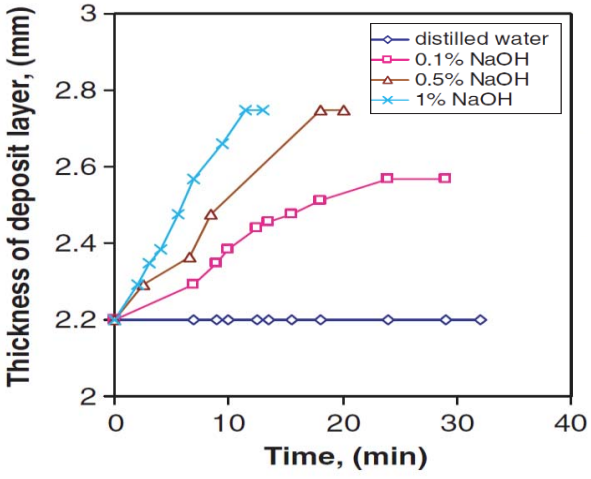
2.3.2 Swelling and Hydration profiles

Swelling of a deposit has been indicated in the literature by two key variables: deposit thickness and deposit mass. Schöler *et al*., (2012) determined deposit thickness from images. The deposit was doped with zinc sulphide. The degree of phosphorescence has been found to correlate with soil thickness. Liu *et al*., (2006, 2007) measured

deposit thickness visually using a side mounted microscope and camera. Figure 1.1 shows the measured thickness for (a) Whey Protein Concentrate (WPC) and (b) egg albumin soaked in distilled water, 0.1, 0.5 and 1 wt% NaOH at 20°C. For both WPC and egg albumin, no increase in deposit thickness is measured in water. Also in both cases an increase in chemical concentration leads to an increase in the maximum deposit thickness achieved and the film growth rates, summarised in Table 1.1 including swelling profile according to Saikhwan *et al*., (2010) classification.



(a)



(b)

Figure 2.7: Deposit thickness vs. soaking time for (a) WPC and (b) egg albumin in distilled water, 0.1, 0.5, 1 wt% NaOH at 20°C

Table 2.2: Swelling profiles for WPC (Liu et al., 2006) and egg albumin (Liu et al., 2007)

Deposit	Concentration NaOH (wt%)	Swelling Type	Maximum thickness (δ_f) mm	time to reach plateau, (t_s) min	film growth rate μm min^{-1}
WPC (containing 0.3 wt% βLg); fouling: 90°C for 120 min	0.1	C	approx. 1.56	5	26
	0.5	B	approx. 2.19	25	58
	1.0	A	approx. 2.88	15	96
Egg albumin (ovalbumin from chicken); fouling 80°C for 60 min.	0.1	B	approx. 2.57	24	15
	0.5	C	approx. 2.75	18	31
	1.0	C	approx. 2.75	11	48

Saikhwan *et al*., (2010) investigated the swelling of protein gels (β-Lactoglobulin (βLg); whey protein isolate (WPI) and WPC at 20°C, using fluid dynamic gauging (FDG) apparatus mounted on a Confocal Laser Scanning Microscope (CLSM); similar to Sahoo *et al*., (2008). They found four distinct swelling behaviours for heat induced protein gels, shown in Figure 2.8

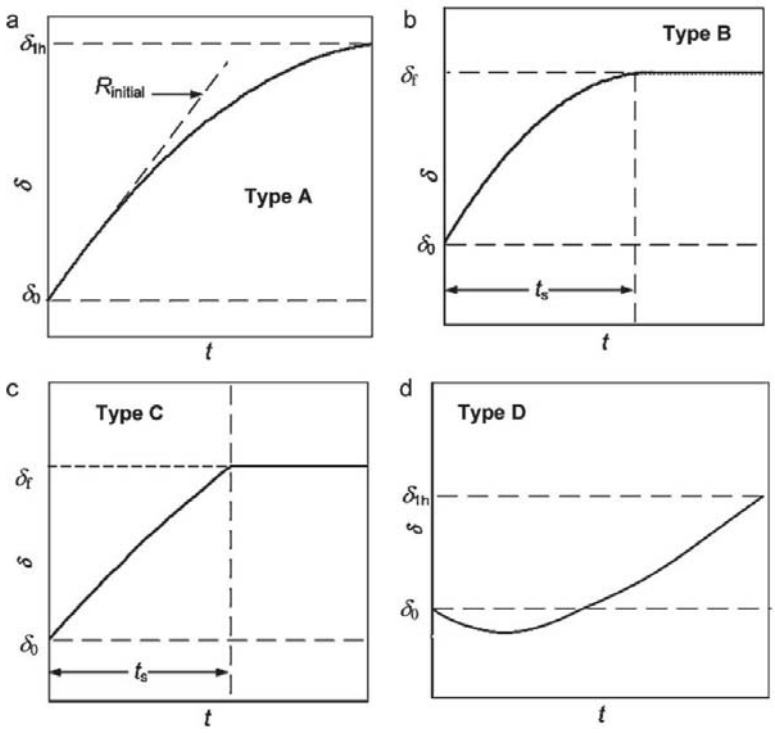


Figure 2.8: Schematic of general swelling behaviour observed for heat induced protein gels (β-Lg, WPI and WPC) at 20°C: (a) neutral pH; (b) pH 12, 0.4M NaCl (salt); (c) pH 12; (d) 1M NaOH (no added salt). Where δ_{1h} is deposit thickness at 1h; t_s is time to reach a swelling plateau (δ_f) and R_{inital} is initial rate of swelling.

The behaviours have been classified and described as:

Type A: βLg deposit swelling in neutral pH, where swelling fit to a Fickian model: $\delta = at^{0.5} + b$ where deposit thickness (δ) rose with the square root of time.

Type B: βLg swelling in 0.4 M NaOH with pH value 12, where similar Fickian increase model is observed but a plateau region after a certain time is reached.

Type C: whey protein concentrate (WPC) swelling in pH value 12.5, where δ was linear for a time before reaching plateau region.

Type D: βLg swelling in 0.1 M NaOH where shrinkage was observed before swelling.

Multiple authors have used FDG to determine deposit thickness. For example, Tuladhar *et al*., (2002) for WPC; Saikhwan *et al*., (2007) for polymer (acrylate-styrene copolymer);

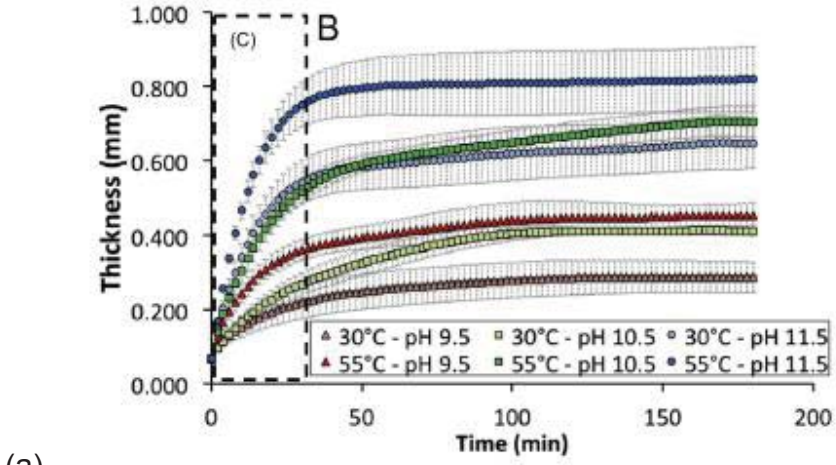
Saikhwan *et al*., (2010) for βLg, WPC and WPI gels. The thickness is measured as a function of mass flowrate through the nozzle (m), the discharge coefficient of the nozzle (C_d), the Re in the siphon tube (Re_t) connected to the nozzle, nozzle inlet diameter (d_t) and the gap between the nozzle inlet and the surface of the deposit film (h) calculated by difference (between nozzle location coordinate and surface location coordinate, using a micrometer). Modified FDG; scanning FDG (sFDG) where multiple locations are measured has also been used by Gordon *et al*., (2010) for gelatin; Pérez-Mohedano *et al*., (2015; 2016) for egg yolk. Figure 1.2 shows the measured thickness of egg yolk in distilled water at 30 or 55°C; pH of 9.5, 10.5 or 11.5 (dependent on salts added) using sFDG. According to Saikhwan *et al*., (2010) classification egg yolk swelling behaviour appear to be Type B for the temperatures and pH tested, with the highest temperature and pH resulting in the largest increase in deposit thickness from approx. 0.1 mm up to 0.8 mm in 40 min (growth rate of 17.5 μm min^{-1}).

2.3.3 Concentration vs. pH vs. temperature on swelling of fouling deposit

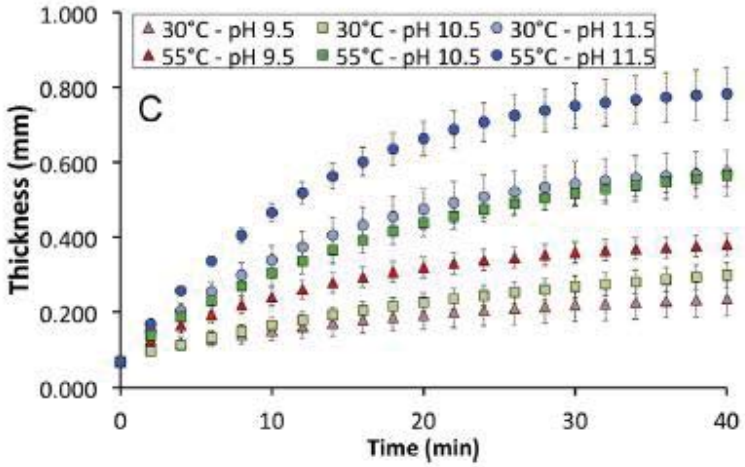
Pérez-Mohedano *et al*., (2016) have used FDG to determine egg yolk deposit thickness (figure 2.9). It is found that increasing in pH and temperature resulted in increasing of swelling and water uptake. The results also indicate that the effect pH is more crucial compare to temperature within the level studied as samples at 30^{o}C and pH 11.5 showed higher deposit thickness than samples at 55^{o}C and pH 10.5. There were some localised blisters found on the on the surface of the egg yolk deposit after 2 hours of experiment at 55^{o}C and pH 11.5 which were believed as the effect of hydrolysis reactions due to high alkalinity. Saikhwan et al., (2010) stated in the previous paper that the high concentration of hydroxyl ions helps to break down the peptide bonds

(cohesive failure) which leading to weakened network strength. Higher in deposit mass and variability after 150 minutes of experiment shown in the gravimetric tests were explained by the presence of big blisters, which help to diffuse the solvent into the network.

Tuladhar et al., (2002) have studied swelling of whey protein film during cleaning using heat flux sensor, fluid dynamic gauge and protein assay in order to measure thermal conductivity, deposit swelling and protein concentration in cleaning solution. In this study the have found that swelling was highly controlled by concentration, pH and flow rate. The swelling stage is longer at low temperatures as the removal of protein might have shortened the swelling stage at higher temperature. Low hydroxide concentrations showed higher swelling ratios but decreased with higher flow rate. At concentration higher than 0.5wt% NaoH, the swelling ratio was not significantly affected by flow rate, while at lower concentration; the swelling ratio was highly affected by flow rate. Results also showed that maximum swelling rate was shown at 0.5 wt% NaoH concentration and swelling rate was significantly lower at concentration less than 1 wt% NaoH for bulk flow condition compared to the swelling rate for stagnant condition. It is believed that the result is strange and unexpected as the shear stress generated by the gauging flow is expected to be higher than the shear stress generated by any other bulk flow condition.



(a)



(b)

Figure 2.9: Deposit thickness vs. hydration time for egg yolk in distilled water at pH 9.5, 10.5, 11.5, at 30 or 55°C (Perez-Mohedano et al., 2016)

2.4 Review on the cleaning of fouling deposit

2.4.1 Dissolution and Cleaning profiles

Deposit mass is normally determined in conjunction with analysis of the cleaning solution to determine dissolution. Dissolution of fouling from the surface into the bulk can be determined in various ways. Typically samples of cleaning solution are collected and the amount of protein present determined using an absorbance technique. For

example:

Modified Bradford Micro-assay with Coomasie Plus Protein Assay Reagent (Gotham *et al*., 2001; Tuladhar *et al*., 2002)

(ii) Modified protein concentration assay method by Kuaye (1994) where the UV absorption difference between 248 and 256 was used to determine protein concentration of the cleaning solution (Xin *et al*., 2002)

2.4.2 Concentration vs. pH vs. temperature vs. flow rate on dissolution and cleaning of fouling deposit

Cleaning is a multistage process (Sandu *et al*., 1985) consists of several steps that controlled by several factors such as diffusion, mass transfer and chemical reaction. Cleaning agents or fluid must be carried to the surface-liquid interface and need to contact and penetrate the deposit (Changani *et al*., 1997). Then, the reaction between the cleaning chemicals and deposit will occur and the removal of the deposit from the surface will take place. The whole cleaning process may be controlled by any stages for example, in high shear area, mass transfer will increase, thus leading to rapid cleaning process (Fryer, 1985). Previous researches on cleaning (Grasshoff, 1989; Bird and Fryer, 1991) indicate that protein deposit swells on contact with sodium hydroxide and that cleaning is not homogeneous but includes uneven removal of lumps of swelled deposit from the surface (Changani *et al*., 1997). If the deposit contains minerals, the acid was would be required to remove the minerals left on the surface. Previous studies also found that the time taken to clean a deposit or foulant is a function of the cleaning chemical concentration, flow rate and temperature (Jennings, 1959). Some researchers

reported that the surface finish, geometry, and the overall process design also affect the cleaning process (Schlussler, 1970; Timperley, 1981).

Generally, the cleaning rate will increase as the temperature increases (Hankinson and Carver, 1968; De Goederen et al., 1989; Fryer and Bird, 1994; Alfa Laval, 2003). Gillham (1999) reported that cleaning of whey protein foulants from stainless steel surface was highly controlled by temperature. Increase in chemical concentration will shorten the cleaning time (Gallot-Lavallee et al.,1984). Plett (1985) has found a linear increase in cleaning rate with increase in cleaning chemical concentration, until it reaches a certain maximum cleaning rate. Some workers have come up with an optimal chemical concentration that helps to decrease cleaning time (De Goederen et al., 1989; Fryer and Bird, 1994; Jeurnink and Brinkmann, 1994). Tissier and lalande (1986) and Belmar-Beiny and Fryer (1993) have studied the activity of the cleaning chemical on the deposit structure. The cleaning fluid motion is needed to transport the cleaning agent to the deposit surface interface, to remove the deposit from the surface and to transport the removed deposit out of the system while the temperature is also crucial as it will help to increase the rate of reaction and the diffusion of cleaning chemicals when the temperature is increase and as well as modify the viscosity of the cleaning chemical fluid (Christian and Fryer, 2003).

2.4.3 The effect of chemical detergents, surfactants and NaOH pH vs. Concentration on dissolution and cleaning of fouling deposit

Type 3 deposit materials need chemicals to make them removable and dissolve them into the cleaning solution (Plett, 1985). Organic based deposits (fats and protein)

required alkaline cleaning chemicals while minerals based deposits required acidic solutions to remove them from the fouling surface. Previous works show that many studied have been carried out to investigate the effect of different concentration of sodium hydroxide (NaOH) on different types of soil material ranging from 0.1% w/w to 5% w/w concentration of sodium hydroxide. They have identified the optimum concentration of sodium hydroxide needed for cleaning depending on the types of soil material used. Bird (1992) found that the optimum concentration of sodium hydroxide for bench scale cleaning of milk and whey protein deposits is 0.5% w/w, while Tuladhar (2001) indicates the optimum concentration of sodium hydroxide for bench scale cleaning of whey protein concentrate (WPC) at temperature 50 ^{o}C and below ranging from 0.5% to 1% w/w. Christian (2003) and Goode *et al*. (2013), believe any concentrations used beyond this optimum concentration will not give a significant effect or benefit and could also lead to longer cleaning time. However, Liu et al. (2007) reports in cleaning of egg albumin, increasing the sodium hydroxide concentration will reduce the cleaning time, but no optimum concentration was found. An optimum concentration needs to be identified as it is not practical to use a high concentration of sodium hydroxide in industry due to its cost.

2.4.4 The effect of Temperature on cleaning of fouling deposit

Increasing the cleaning temperature will increase the solubility of fouling deposit into cleaning agent, the reaction of soil towards the cleaning solution and rate of diffusion, possible reduction of yield stress, and viscosity of fouling deposit which accommodates cleaning and weaken the adhesive and cohesive strength of the fouling deposits

(Alharti, 2013; Palabiyik, 2013). Liu *et al*. (2007) indicates that increasing soaking solution temperature will reduce the apparent adhesive strength of egg albumin deposits measured by micromanipulation technique. Christian (2003) suggests that temperature plays more important role in cleaning than flow velocity. The effect of cleaning temperature has been studied in previous works which ranging from 13^{o}C to 90^{o}C depending on the type of soil. Gallot-Lavalle *et al*. (1984) stated that the optimum cleaning temperature for proteinaceous deposits is 65^{o}C while Hankinson and Carver (1968) found that 55^{o}C is the optimum cleaning temperature for milk deposits cleaning with water. An optimum temperature for cleaning is much needed as it will affect the cost of energy consumed.

2.4.5 Balance between chemical action and mechanical action in cleaning

Both chemical action and mechanical action are required in order to create an effective system to clean the deposit from the surface. Literally, these chemical and mechanical actions were believed to come from the cleaning fluid used (*Changani et al*., 1997*; Fryer et al*., 2006; *Cole*, 2013).

The balance between chemical and mechanical cleaning must be assessed and if a soil needs chemical breakdown to allow removal, a sufficient amount of chemical must be applied and given some time to act on the deposit (*Cole*, 2013). *Gallot-Lavallee* (1984) has studied cleaning kinetics of milk deposit on a tubular heat exchanger at a pilot plant scale and came up with a 2 stage model of cleaning kinetics; zero order equation which represents the chemical action of the milk deposit and first order equation which represents the mechanical action of the cleaning fluid flow.

Another researcher *Visser* (1995) reported that wall shear stress was found to play a major role in the removal of colloid particles but the presence of chemical fluid (cleaning agent) helps to weaken the adhesive force between the deposit and the surface and thus increase the removal of colloid particles. These findings show that the combination of mechanical and chemical action is essential in order to create an effective cleaning system.

Bird and fryer (1991) identified three major process involved in *chemical cleaning process*;

i) Mass transfer; removal of deposit by fluid dynamics which believed to be the most effective cleaning mechanism.

ii) Diffusion; an essential cleaning mechanism for deposit which can be removed by hydration process. *Plett* (1985) reported that diffusion helps to bring the chemical into the deposit structure.

iii) Reaction processes; these processes involved in cleaning of materials which require chemical breakdown. *Plett* (1985) explains how bulk reaction dominates the cleaning process.

Several researches have carried out studies to understand the cleaning mechanism. *Morison* and *Thorpe* (2002) observed the effects of interfacial tension between the cleaning fluid and the fouling material using spinning disc technologies which control mass transfer effects. Following this observation, they came up with a five step mechanism of removal reaction.

i) *Mass transfer of chemical into the boundary layer*. Chemical concentration and

cleaning fluid flow rate were found to be the key player to control the transfer rate and the width of the boundary layer.

ii) *Interfacial process through the fouling material* which depends on cleaning fluid concentration.

iii) *Interfacial process to the unreacted fouling material* which also depends on cleaning fluid concentration.

iv) *Interfacial process* which depends on the solution (protein release), concentration of cleaning fluid and nature of cleaning fluid.

v) *Mass transfer of the protein through the boundary layer*. Chemical concentration and solution flow rate were found to control the transfer rate and the width of the boundary layer.

A part from the chemical action of cleaning, previous researchers have also carried out studies to investigate the mechanical action of cleaning. *Bird* and *Fryer* (1991) found that flow rates affect different phases in a foulant removal differently. They also suggested that increase in flow rates would increase the Reynolds number of the fluid and thus, reduce the cleaning time. *De Goederen et al* (1989) also stated that Increase in Reynolds number of the fluid may ease the removal of the deposit. The mechanical effects of the fluid flow may help in further removal of a fouling material after being exposed to fluid flow. Other researches such as *Timperley* and *Smeulders* (1988) also reported that increase in flow rate from 0.2 ms^{-1} to 1.5 ms^{-1} led to quicker deposits removal of a plate heat exchanger. *Othman et al* (2010) conducted a research to study the cleaning of sweet condensed milk (SCM) fouling deposit on stainless steel surface using a flow cell which found that increasing the flow rate of chemical fluid from 0.25 to

0.5 ms^{-1} led to quicker deposit removal at all temperatures. *Christian* (2004) suggested that higher flow speed may produce greater shear stress on the fouling material and reduce the boundary layer which consequently reduce the cleaning time of a fouling material. *Klavenes et al* (2002) conducted a study to determine adhesion by measuring a range of shear stresses using a radial flow cell. A study by Visser (1995) found that surface condition affect the removal of fouling material from the surface. It was also found that surface hydration and steric hindrance play an important role in reducing the adhesive force by preventing the close approach of fouling material and surface. *Bott* (1995) stated that *Van der Waals* forces, electrostatic forces, hydrogen-bonding and hydrophobic bonding was believed to be the major forces that create adhesion between surface and deposit. *Liu et al* (2002) carried out a research to study the adhesion force between tomato paste deposit and stainless steel surface using a micromanipulator. It has come into a conclusion that hydration of tomato paste deposit in cleaning fluid helps to weaken the adhesive force strength between surface and deposit which consequently ease the deposit removal. *Akhtar et al* (2010) conducted a study to investigate adhesive forces of different fouling materials (toothpaste, sweet condensed milk, turkish delight, and caramel) fouled on different surfaces (glass, stainless steel and PTFE substrate) using Atomic Force Microscopy (AFM) which found that caramel had the greatest adhesive force (0.4 Nm^{-1}) while toothpaste had the smallest adhesive force (less than 0.005 Nm^{-1}).

In this research, egg yolk is used as a type 3 foulant material. Chicken egg has been widely used as an ingredient in food manufacturing industry. However, fouling occurs because of egg yolk ability to coagulate easily at high temperature during thermal

treatment in industry which leads to formation of highly elastic gel-like deposit on the surface (*Paraskevopoulou and Kiosseoglou*, 1997). If there is no proper cleaning system established during the manufacturing process, this formation of egg yolk deposit can be a nutrient source to microorganisms which leads to food contamination and degradation of food processing equipment performance (*Hagiwara et al*., 2015) due to High Density Lipoproteins (HDLs) content in egg yolk which can only be broken down by a high ionic strength media like sodium hydroxide because of their complex structure and the bonds created between them (*Anton and Gandermer*, 1997). To make it worse, this fouling build-up will increase the surface roughness and decline the cross-sectional area of flow channels in pipes or heat exchangers which increases the pressure drop in the thermal system and declines the heat transfer efficiency in the thermal system (*Paul et la*., 2014).

As a type 3 fouling material, a large amount of water, chemicals, energy and time required for egg yolk deposit to be cleaned up (*Christian et al*., 2002). *Cole* (2011) indicates that it is well understood that hydration helps a foulant to be removed from its surface. *Liu et al*. (2003) has also reported that the adhesive force between the deposit and the surface was decrease when the samples soaked in cleaning solutions prior to measurement on the micromanipulation rig. Research by *Fryer et* al. (2006) reported that hydration of deposit was found to be a key mechanism for deposit removal.

2.5 Review on the method development and previous works

2.5.1 The development of micromanipulation technique

The adhesion of the deposits and the surface, the cohesion of the deposits material and the shear force applied are known to be the main theories behind the development of micromanipulation technique by (*Liu et al*., 2002). Adhesion is the tendency of dissimilar particles to bond to on another while cohesion is the tendency of similar or identical particles, surfaces or materials to bond one another (*Fraunhofer*, 2011). Shear force can be described as an internal force in any material which is usually caused by any external force acting perpendicular to the material, or a force which has a component acting tangent to the material. The shear force may vary at different point in the material. In this micromanipulation technique, a T-shaped probe has been used to apply shear force on the food fouling deposits was studied in terms of the apparent adhesion strength of the deposits and the surface, and the cohesion strength between the deposits material (*Liu et al*., 2002). The force was determined by a force transducer connected to the probe and this technique allows deposit layers to be removed at millimeter level (*Liu et al*., 2002). This method was firstly developed by *Chen, Zhang* and *Bott* (1998) to study biofilms, however, it has been extended to study various food films.

2.5.2 Micromanipulation rig set-up

Liu et al (2002) developed a technique to measure the force required to remove a deposit from a surface using a T-shaped probe as tool to provide shear force to the deposit. This micromanipulator consists of a T-shaped probe which connected to a

force transducer (Model BG-1000, Kulite semiconductor, Leonia, NJ, USA). Sample was placed on a stage (sample base) that attached to a micromanipulator (MicroInstruments, Oxon, UK). This micromanipulator holds microscopes which connected to the monitors that provide top and side view. A computer was used to control the operation and was connected to a multifunctional data acquisition board (Amplicon Liveline, Brighton, UK) which was used to record the force exerted on the probe at 100 Hz during the experiment (Liu *et al*., 2007). The gap between probe and the surface can be adjusted through the side view monitor and the whole operation can be monitored through the top view monitor. The transducer needs to be calibrated prior to the experiment. Data collected from the data acquisition board was saved and analysed in the computer.

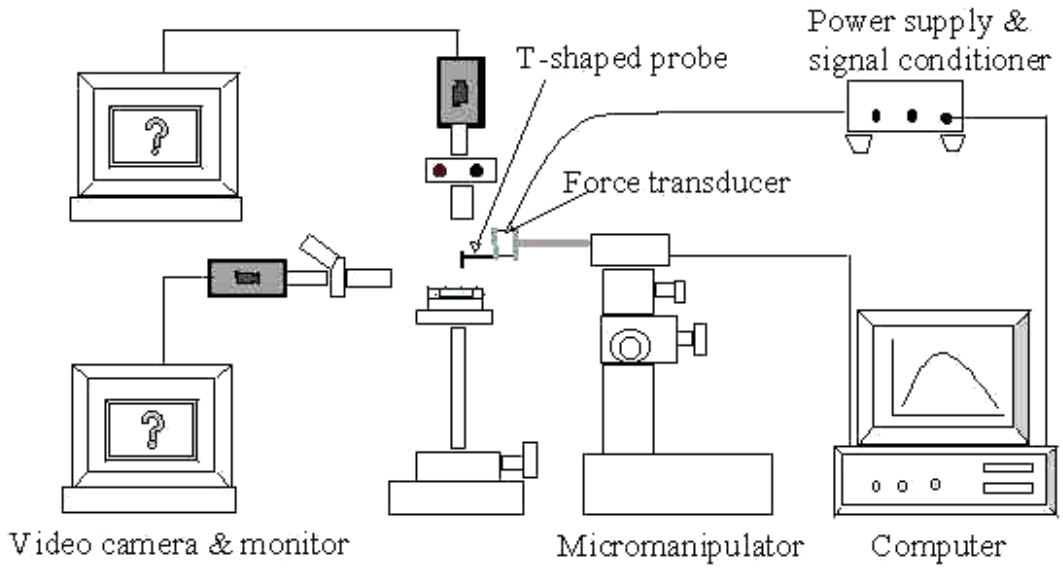


Figure 2.10: Schematic of the micromanipulation rig, based at the *University of Birmingham* by (Liu et al., 2002)

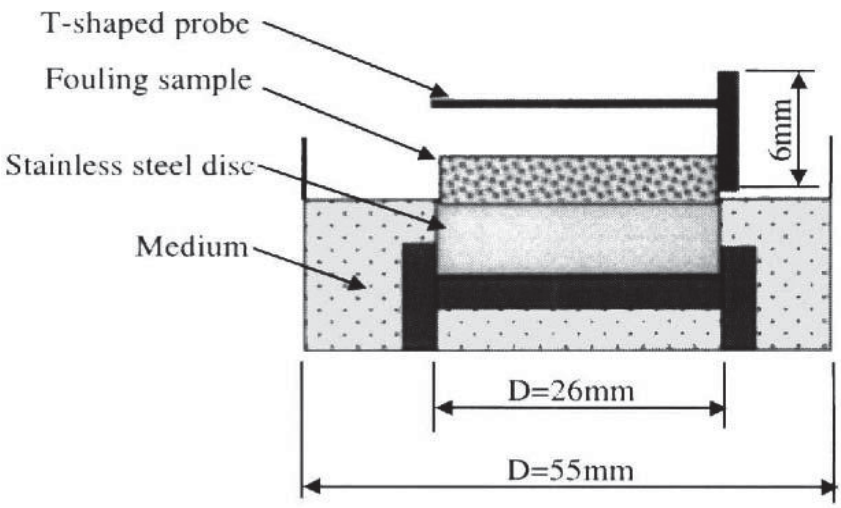


Figure 2.11: Schematic of the T-shaped probe, fouling sample, and stainless steel disc by (Liu *et al*., 2002)

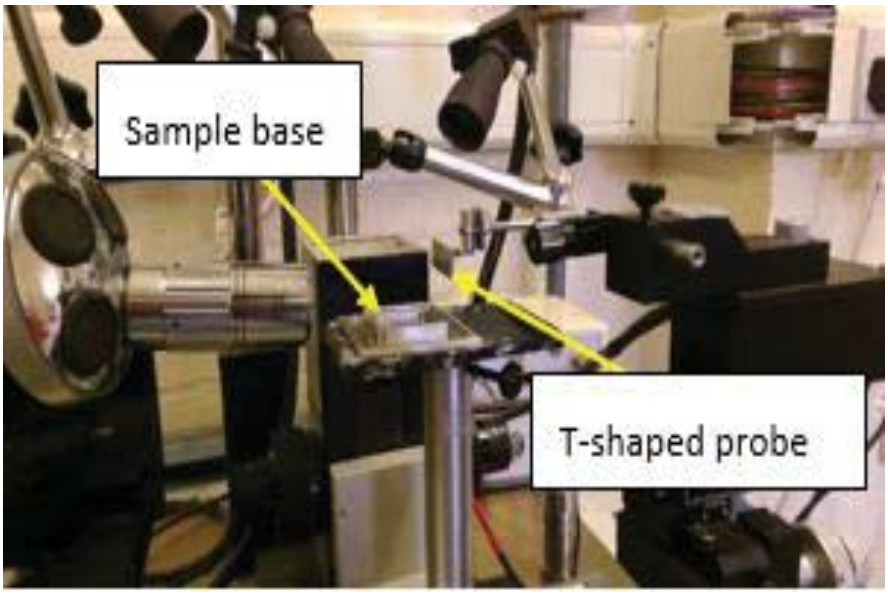


Figure 2.12: The sample base and the T-shaped probe on the micromanipulation rig

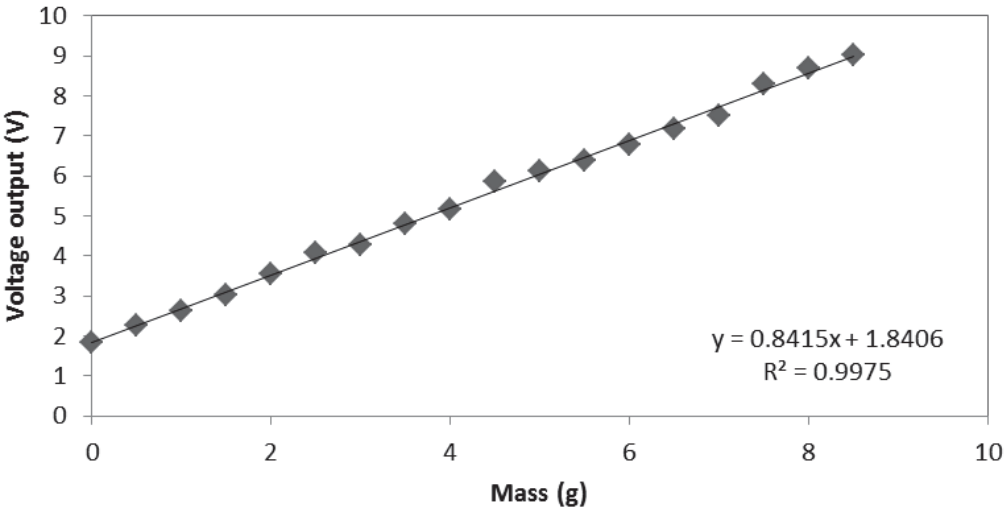


Figure 2.13: Calibration of the 10 g force transducer by Goode (2012)

The force required to remove the deposit was measured by moving the micromanipulation arm across the surface of the deposit. Figure 2.8 illustrates the typical force vs sampling time curve derived from the micromanipulation data. The sample was pulled horizontally from the initial contact point A, while point B and C represents the center of the disc and the final edge. During experiment, the force measured by the transducer was increased to a maximum point from A to B, which corresponds to the maximum width of the deposit and then decreased to the lowest point from B to C). The saw-tooth shape of the force curve was derived from the deformation and detachment of soil during pulling (Liu et al., 2002). The deposit was pulled from the surface in one piece. From point C to D, No significant force was applied on the probe as the sample was pulled away from the surface (Liu et al., 2002).

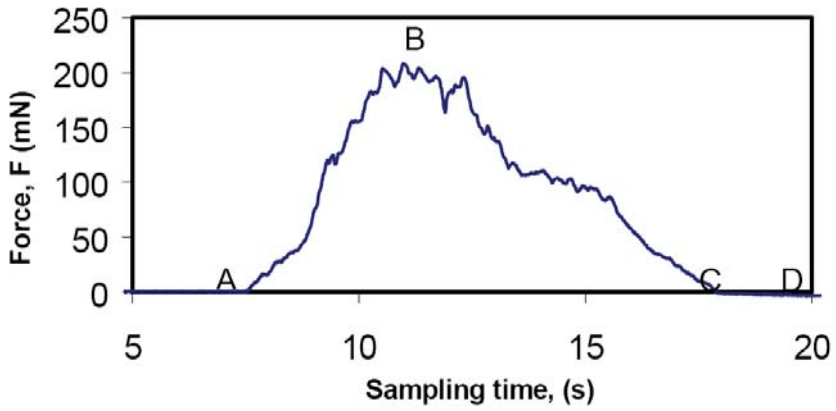


Figure 2.14: Typical force curve vs. sampling time on the micromanipulation rig for baked tomato paste removal by Liu et al., (2003)

The force measured depended on the gap between the probe and the surface which determine the cut height of the probe through the sample. At higher cut height, deposit was not totally removed which means a layer of deposit left after the experiment. In this case, the force measured was the cohesive force between the elements in the sample. The adhesive force between the sample and the surface was measured when the sample was totally removed from the surface (Cole, 2013).

Data analysis was carried out according to the method developed by Liu *et al*. (2002). The data collected from the data acquisition board for each experiment was in the unit of volt. The data in volt was multiplied a factor in order to change the unit from volt to force (mN).

Calibration factor = transducer value (slope of the calibration curve) x 9.8

Force = voltage x calibration factor

It is based on 1 kg force = 9.8 N or 1 g force = 9.8 mN

The total work, W (J) done by applied force, F(t) to remove the deposit was calculated as the integral of

$$dW = Fdx \quad (2)$$

Where the distance d*x* is *v* d*t*, so that

$$W = d/(t_c - t_a)\int_{ta}^{tc} F\,dt \quad (3)$$

Where *d* is the diameter of the rectangular coupon, t_a and t_c is the first and the last times at which the probe touched the fouled surface.

The apparent adhesive strength of a fouling sample, σ (J/m^2) defined as the work required to remove the sample per unit from the surface to which it is attached is then given by

$$\sigma = \frac{W}{\alpha A} \quad (4)$$

Where A (m^2) is the surface area, and α is the fraction of that area covered by the sample measured, in which α has been taken as 100 % in this study because the samples were totally removed by the micromanipulation probe.

In this study, micromanipulation will be used to investigate the effect of different pre-soaking conditions i.e. temperature and time on the adhesive and cohesive force of egg yolk deposit.

2.5.3 Review of the previous works using micromanipulation technique

Liu et al. (2002) has studied the effect of experimental variables using micromanipulation technique which tomato and whey protein have been used as samples. The way in which the deposits removed is different in both cases. Tomato deposits are removed by applying a shear force to overcome forces between surface and deposits, while in whey protein case, a shear force is applied in order to resist forces between the deposits itself and between surface and deposits. Results in whey

protein experiments show that the apparent adhesive strength is affected by temperature, cleaning agent concentration, and exposure time while in tomato deposits experiments, it shows that the apparent adhesive strength is affected by surface roughness, sample heating time, the thickness of sample, and sample hydration time. Experiments with whey protein samples result in greater adhesive strength compare to the cohesive strength while in the case of tomato deposits, the adhesive strength is smaller than the cohesive strength (*Liu et al*., 2002).

Studies by Liu et al (2006) using this to technique to investigate force needed to remove whey protein concentration (WPC) deposit from stainless steel surface found that the adhesive force between the WPC deposit and the surface and the cohesive force within the deposit structure were controlled by temperature, cleaning chemical concentration, and hydration time. At room temperature, the adhesive force of WPC deposit was higher than the cohesive. The adhesive and cohesive force of WPC deposit was found to be between 0 and 10 Jm^{-2}. *Liu et al*. (2007) described that both cohesive and adhesive strength are affected by experimental variables in which the experiments with ovalbumin deposits result in greater cohesive strength as compared to the adhesive strength at room temperature. It is also stated that this method can be used to study cleaning by observing the effect of cleaning agent concentration on cleaning time. *Liu et al*. (2006b) has come out with a simple model to understand the mechanisms of food deposits removal. At low height of the probe, *x,* the deposits break away from the surface and this is affected by the surface properties, while at high *x,* the deposits removed by the bonding breakage between the deposits material itself which resulted in a layer of deposits left on the surface. This phenomenon is not affected by the surface

properties and the force required removing the material decreases with the probe height (*Liu et al*., 2006b).

Cole (2011) has also conducted studies to investigate the adhesive and cohesive force of toothpaste deposit using this micromanipulation technique. It was found that the adhesive force (61.5 $\pm$ 2.6 Jm^{-2}) was greater that the cohesive force (44.6 $\pm$ 1.2 Jm^{-2}) of toothpaste. Pre-soaking of deposit for 60 s at 30^{o}C helped to decrease the adhesive force of toothpaste by 30%. Cole (2011) also found no strong relationship between the previous results of various deposits studied using cleaning rig and the previous results of various deposits using micromanipulation technique.

Akhtar (2010) used micromanipulation technique to study the pulling energy of caramel, toothpaste, sweet condensed milk (SCM) and Turkish delight deposit on stainless steel, PTFE and glass surface. For caramel deposit, it was found that increasing the probed height increased the pulling energy, while for toothpaste, SCM and Turkish delight deposits, increasing the probe height reduced the pulling energy. For caramel, SCM, and toothpaste deposit, stainless steel gave the highest pulling energy followed by glass and PTFE while for Turkish delight deposit, the difference in pulling energy between stainless steel, glass and PTFE surface is not significant. Goode (2012) also used this technique to characterize the yeast slurry which found that the adhesive strength was higher than the cohesive strength of the yeast slurry deposit fouled on stainless steel surface.

Rois Anwar (2016) used the similar technique to study the effect of different hydration fluid, hydration time, hydration temperature, materials, probe speed, tempering and cooling on the adhesive and cohesive force of chocolate deposit on polycarbonate,

PTFE and stainless steel surface. Chocolate deposits would not be removed without pre-soaking treatment and in this case, sodium hydroxide (0.1% w/t) has been used in order to remove the deposit. Increase in pre-soaking temperature and time led to decrease in pulling energy of chocolate deposit. Stainless gave the lowest pulling energy required to remove the deposit, followed by polycarbonate and PTFE. No complete removal was observed in all cases as a thin chocolate layer was seen left on the surface. It was found that probe speed between 0.59 and 2.2mms^{-1} did not affect the pulling energy of the chocolate deposit.

2.5.4 The Development of cleaning rig

A flat plate flow cell rig was developed by Kostantina Asteriadou at the University of Birmingham in order to study the cleaning of fouling deposit at lab scale (Christian, 2004). This rig consists of a rectangular flow channel with test section where the fouled coupon sample is placed, a heating tank to regulate the temperature, a reservoir tank to store water or chemical used for cleaning, a centrifugal pump to circulate the water flow, a flow transmitter 340 D to regulate the flow rate, thermocouples as the temperature probe, a microfoil heat flux sensor (MHFS) unit, a data logger system to record the data from the thermocouples, flow transmitter and The MHFS unit and a computer used to save the data and control the operation.

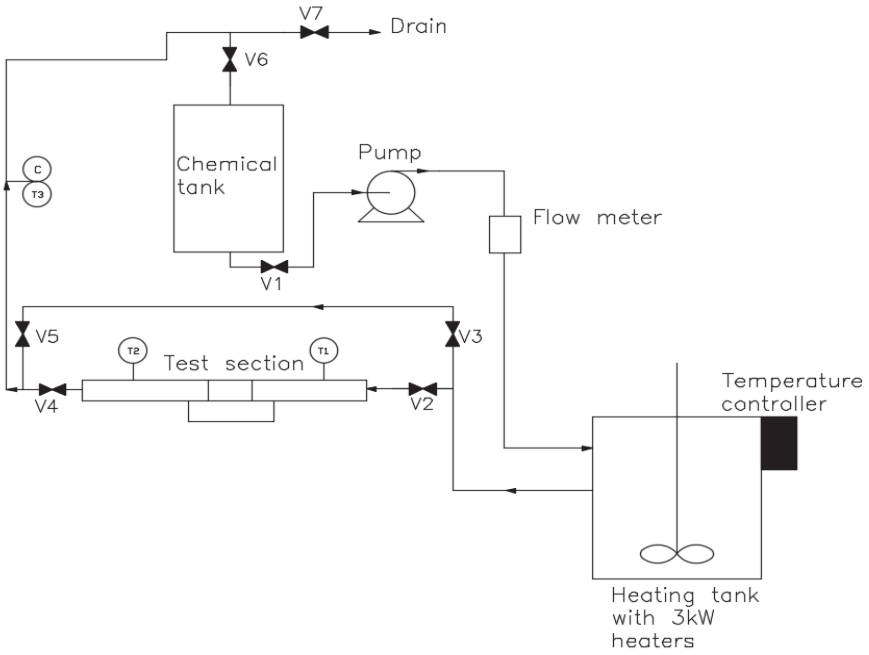


Figure 2.15: Schematic diagram of the flat plate flow cell rig by (Palabiyik et al., 2014) and (Christian and Fryer, 2006)

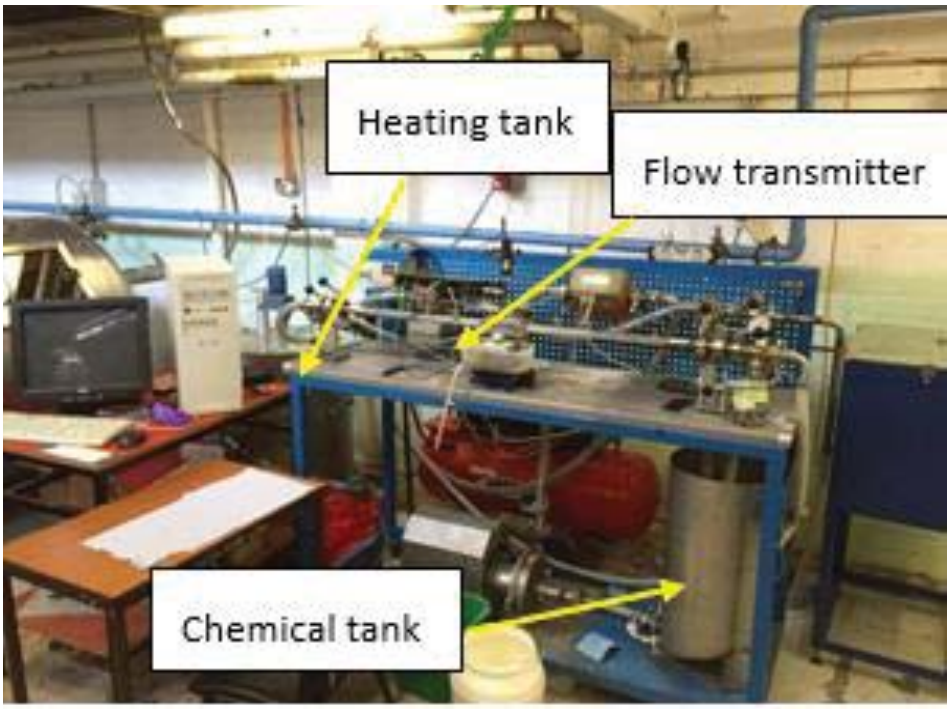


Figure 2.16: The flat plate flow cell rig set-up

The fouled coupon sample was slotted in the test section which was sat on the heat flux sensor unit to make sure that the bottom side of the coupon touches the cooper disk of the heat flux unit. The deposit was removed by the water flow circulated from the reservoir tank. The test section was designed so that the deposit removal could be monitored from the side and top view using camera. The MHFS unit was placed in the ice box to provide a maximum temperature difference between the heat flux sensor and the cleaning fluid. Heat flux was created from the temperature of the cleaning fluid insulated by the deposit and the surface placed right above the heat flux unit (Cole, 2011). As the deposit was removed gradually from the surface during the experiment, the heat transfer between the cleaning fluid and the heat flux sensor unit would increase. This changes in heat flux could be used to calculate the overall heat transfer coefficient, U (kWm^{-2}K^{-1}) using the following equation;

$$U = \frac{q}{T_L - T_{HB}} \tag{5}$$

Where T_L is the average temperature of the cleaning fluids giving by the thermocouples, T_{HB} is the temperature of the copper disk and q (kWm^{-2}) is the heat flux across the sensor given by the following equation (Cole, 2011);

$$q = \frac{\lambda_s}{x_s}.\varphi.KT_B.V \tag{6}$$

Where λ_s (kWm^{-1}K^{-1}) is the sensor thermal conductivity, x_s (m) is the thickness of the sensor, φ is a constant (V^{-1}), KT$_B$ is a dimensionless factor, and V is the voltage reading from the MHFS.

The following equation was derived from a linear relationship between K.T$_B$ and temperature based on the calibration chart of the MHFS given by RdF (MHFS supplier);

$$K = -0.001T_B + 1.0274 \quad (7)$$

The value of T$_B$ was approximated to be T$_{C2}$ (Goode, 2012). RdF gave the value for the factor $\frac{\alpha_s}{x_s}.\varphi$ as follows;

$$\frac{\alpha_s}{x_s}.\varphi = 11.9\ \mu V\ Btu^{-1}ft^{-2}hr^{-1} \quad (8)$$

Thus, by converting Btu ft^2.h.μV^{-1} to S.I unit (11.9 multiplied by 3.154591 x 10^{-3}), the heat flux across the sensor q (kWm^{-2}) could be calculated as follows;

$$q = 0.000265.(-0.001T_{C2} + 1.0274).V \quad (9)$$

Therefore, the heat transfer coefficient U (kWm^{-2}K^{-1}) could be calculated using the following equation;

$$U = \frac{0.000265.(-0.001T_{C2} + 1.0274).V}{T_L - T_{C2}} \quad (10)$$

Where T_{C2} is the temperature of the copper disk, T_L is the average temperature of the cleaning fluids giving by the thermocouples, and V is the voltage reading of the MHFS.

Removal phase time could be determined by deposit resistance, R_d (Goode, 2012) using the following equation;

$$R_d = \frac{1}{U_t} - \frac{1}{U_c} \quad (11)$$

Where U_t is the heat transfer coefficient at time t and U_c is the heat transfer coefficient of clean coupon.

2.5.5 Review of previous works using cleaning rig

Cleaning rig has been used as an established method by the previous researchers to the study cleaning of fouling materials at lab scale. Christian (2004) conducted a research to investigate the cleaning of tomato paste and whey protein concentration (WPC) deposit. A linear relationship was found between increase in temperature and decrease in cleaning time of tomato paste deposit. Cleaning time was minimised by increasing the temperature from 30 to 70^{o}C and flow rate from 0.7 to 2.3 l min^{-1}. Increasing the temperature and flow velocity from the lowest temperature and flow velocity to the highest temperature and flow velocity shortened the cleaning time by 6 times. For the cleaning of WPC deposit, researcher found no deposit removal at all

temperatures (30 – 70^{o}C) and flow rates (0.7 to 2.3 l min^{-1}) used without chemical action. At 0.5% caustic solution, increase in temperature and flow velocity gave shorter cleaning time. Christian (2004) concluded that the effect of temperature is greater than flow rate and chemical concentration in the cleaning of WPC deposit.

Aziz (2008) also carried a study a study to investigate the cleaning behaviour of egg albumin gel using the cleaning rig (coupon rig). No deposit removal was found without chemical cleaning. Temperature plays a major role in the cleaning of egg albumin deposit as it can affect the strength of chemical concentration and flow velocity effect. Increase in temperature decreased the cleaning time. However, the shortest cleaning time did not achieved by increasing all the experimental factors. Significant decrease in cleaning time was normally shown at initial increase in chemical concentration and flow velocity. There was no optimum chemical concentration at 50^{o}C and 1.5 l min^{-1} flow velocity. Generally, increase in chemical concentration decreased the cleaning time.

Othman et al (2010) used a flow cell to characterise the cleaning behaviour of sweet condensed milk (SCM) fouled on stainless steel coupon. Generally, they found that flow rate, chemical concentration and temperature affected the cleaning behaviour of SCM. At all temperatures tested, the cleaning was reduced by increasing the flow rate from 0.25 to 0.5 m s^{-1}. A linear relationship was found between increase in temperature from 40 to 80^{o}C and decrease in cleaning time. However, no significant effect was found by increasing the caustic solution concentration from 0.5 to 1.5 %. Therefore the authors concluded that temperature had greater effect than flow velocity and chemical concentration on the cleaning of SCM deposit (Goode, 2012).

A study by Palabiyik (2013) used the similar method to study the effect of the product recovery stage on the cleaning of pipes at lab scale with toothpaste was being used as a fouling material. The author found that increase in flow rate of water reduced the cleaning time at ambient temperature and increase in temperature at all flow rates reduced the cleaning time. Cleaning of toothpaste is a function of physical action as no chemical needed to remove the deposit.

In the study of Cole (2011), coupon rig was used in order to study the cleaning of tooth paste at lab scale which found that increase in flow rate and temperature helped to minimise the cleaning time. Complete removal was achieved with flow velocity and temperature and no chemical needed for this cleaning. Therefore, it was concluded that toothpaste is classified as a type 1 deposit.

Goode (2012) used this cleaning rig to study the effect of different flow rates, temperatures and chemical concentrations on the cleaning of yeast slurry, aged yeast slurry and cooked caramel deposit fouled on stainless steel coupon. It was found that complete removal of yeast slurry deposit was achieved by cleaning with water at all flow rates and temperatures. The effect of different temperatures on cleaning appeared to be less significant and increase in flow rate decreased the cleaning time of yeast slurry deposit. Therefore, it was concluded that yeast is a type 1 deposit as it did not require chemical action to be removed. For the cleaning of aged yeast slurry deposit, it was found that complete deposit removal was not achieved by cleaning with water. Flow rate showed no significant effect except at high temperature and temperature helped to reduce the lag phase time and seemed to reduce the removal phase time. Complete removal of aged yeast slurry was achieved by chemical cleaning with the effect of

temperature and chemical concentration was clearly seen at low flow rate while at higher flow rate, the effect of temperature was greater than the effect of chemical concentration. For the cleaning of cooked caramel deposit, it was found that complete removal was only achieved at 80oC. The effect of flow rate and temperature was greater than the effect of chemical concentration on the deposit removal of cooked caramel deposit.

In the study by Rois Anwar (2016), cleaning of chocolate deposit on stainless steel, polycarbonate and PTFE surface was investigated using cleaning rig. No complete deposit removal was seen without chemical action even at the highest temperature ($70^{o}C$) and flow rate (0.5 ms^{-1}). At 0.1% caustic solution, complete removal of deposit fouled on polycarbonate was found at 40, 55 and $70^{o}C$. Increasing the flow rate from 0.25 to 0.37 ms-1 reduced the cleaning time by 20 %, while increasing the flow from 0.37 to 0.5 ms-1 decreased the cleaning time by 60 %. PTFE was found to have the longest cleaning time compares to polycarbonate and stainless steel.

In this study, this cleaning rig will be used to study the effect of different concentrations of sodium hydroxide and temperatures at 0.5 ms-1 on the cleaning of egg yolk deposit.

2.5.6 Recent development in cleaning of food fouling deposit

Vicaria et al (2018) used electrocleaning to study the cleaning of food deposits (starch and heat-denatured whey protein) on stainless steel surface. Anionic and non-ionic surfactants were used to study the effectiveness of the cleaning. It was found that the detergency of the surfactants was improved when starch soil was used. However, this method did not substantially improve the surfactant detergency when denatured whey

protein soil was used. Fan et al (2018) conducted a research to investigate the effect of Reynolds number, rinsing temperature and rinsing time on water effectiveness and efficiency in the cleaning of skim milk residue film from stainless steel pipe surfaces. Results from the experiments indicated that the effectiveness of rinse water increased as the Re increased, but not in linear proportionality. Rinse water efficiency was found to be decreased with increased rinse water volume and rinse time. Rinsing at more than 45^{o}C gives limited impact on water rinsing.

Zhang et al (2018) carried out a research to study the effect of glucose, sucrose and fructose on the fouling behaviour of whey protein using model solutions consisting of whey protein isolate (WPI) and sugars in a spinning disk apparatus under well-controlled temperatures and shear stresses. The addition of 10% of sugar (wt) resulted in reduction of WPI fouling by more than 30% and also affected the composition and the morphology of the deposits, which had lower protein content and higher porosity. This reduction of WPI fouling was identified due to the stabilizing effect of sugar on WPI. Increasing shear stress also reduced the fouling of WPI.

Sauk et al (2018) has developed an online measurement of particles detached from a starch fouling layer during a cleaning process. The automated particle analyser for cleaning (APAC) was designed by combining a flow channel with a laser diffraction particle size analyser. This method could be used to monitor cleaning progress and behaviour. It was found that a narrow particle size distribution (PSD) of small particles suggested a cohesive failure and a steady disintegration of the fouling layer from top to bottom while a broader PSD shape of larger size particles indicated an adhesive failure and detachment of large patches of the fouling layer.

CHAPTER 3: MATERIALS AND METHODS

3.1 Materials

3.1.1 Surfaces

Stainless steel (AIS 316) coupons 2 mm thick were used as the surface for all food deposit samples. The coupons were 30 mm x 30 mm, with a milled ridge (2.5 mm x 2.5 mm) at the outer edge to fix into the test section base of the cleaning rig. . The average surface roughness (S_a) was measured from R_a profiles of a 5 mm line on three random locations on each coupon (R_a of 0.391 μm ± 0.034) using the Talysurf 120L (Rank Taylor Hobson, USA). The contact angle of a water droplet on each coupon was determined by sessile drop technique (using approximately 25 μL of deionized water) and was measured (66 ± 4°) from recorded images using a CCD camera. The upper face of each coupon (area 625 mm^2) is fouled and in contact with cleaning fluid or the probe during each experiment.

3.1.2 Egg yolk composition

The approximate wt% of components in egg yolk powder (E0625, Sigma-Aldrich, UK) is approx. 62.5% fat, 33% protein, 1% carbohydrates and less than 3.5% mineral (Mine and Zhang, 2013). The association of fat and protein in egg yolk is high. All lipids in egg yolk are found to be associated in lipoprotein complexes. The fat component comprises 62% triglyceride, 33% phospholipid, < 5% cholesterol. Carotenoids, which give egg yolk its colour represent < 1% of the total lipid amount (*Fredriksson et al*., 2006). Egg yolk powder (5 g) was mixed by stirring using magnetic stirrer for 15 minutes at room

temperature with water (8 mL) to create an egg yolk paste that could be applied to coupons.

3.1.3 Fouling procedure

Egg yolk paste (1 g ±0.05 g) was spread onto each coupon face (using a disposable plastic pipette) and placed in a convention oven (Gallenkamp CHF097 lab oven) for 60 min at 90°C. The samples were cooled down to room temperature (taking approx. 2 hours), and the deposit mass recorded prior to the experiments. An example of the fouling deposit is shown in Figure 3.1.

Figure 3.1 Egg yolk deposit sample fouled on a stainless steel coupon

3.2 Methods

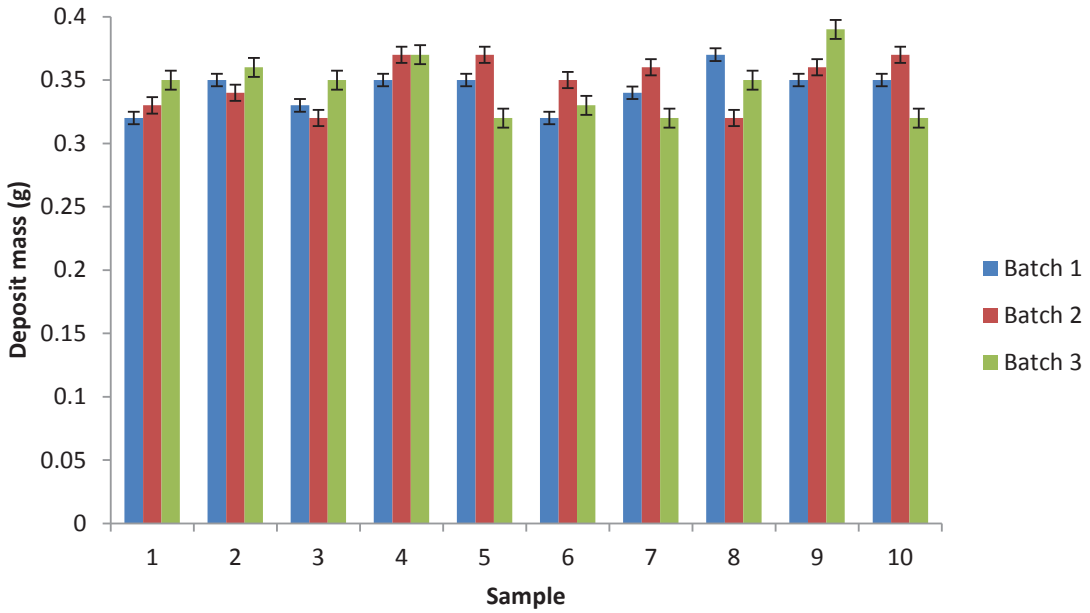
3.2.1 Deposit reproducibility

This analysis was carried out in order to get high reproducibility of egg yolk deposit sample and to make sure that variation of results obtained from the experiments is due to the manipulation of the experimental parameters (chemical concentrations, temperatures, stirring speed etc.) but not because of the variability of the sample itself. Deposit mass, thickness and heating homogeneity in the oven may affect deposit reproducibility.

Each stainless steel coupon mass was measured before and after baking to get the initial mass deposit. The deposit thickness was measured using ImageJ software as described in section 3.2.3. However, due to uneven temperature distribution in the oven, it is hard to ensure the heating homogeneity within the oven. 15 to 20 samples are baked in each batch and only the best 10 samples are selected from each batch to be used in the experiments. The selection is done based on deposit colour uniformity after baking.

Figure 3.2(a) represents the initial deposit mass measured prior to experiments, while Figure 3.2(b) shows the initial deposit thickness prior to experiments. Based on these two diagrams, samples are not uniform in terms of deposit mass and thickness between batches and also within the same batch. This non-uniformity is thought to be due to the variation in the amount of sample pipetted (1 $\pm$ 0.05g) onto the stainless steel surface during samples preparation and the low accuracy of the disposable pipettes, in which variation of 8.57% within the mean value is obtained for deposit mass and 8.33% for deposit thickness. Non-uniformity also might be caused by the uneven heat and temperature distribution within the oven which causes variation in the rate of evaporation of sample during baking of egg yolk. Based on the data collected, the average and the standard deviation of the deposit mass is 0.37 $\pm$ 0.03g, while 1.3 $\pm$ 0.1mm for deposit thickness. The 8.49% and 8.29% variation is considered to be acceptable as slightly higher than the variation percentage (8%) obtained by *Liu et al*. (2006) when preparing egg albumin deposit on the similar type of stainless steel coupon.

a)



b)

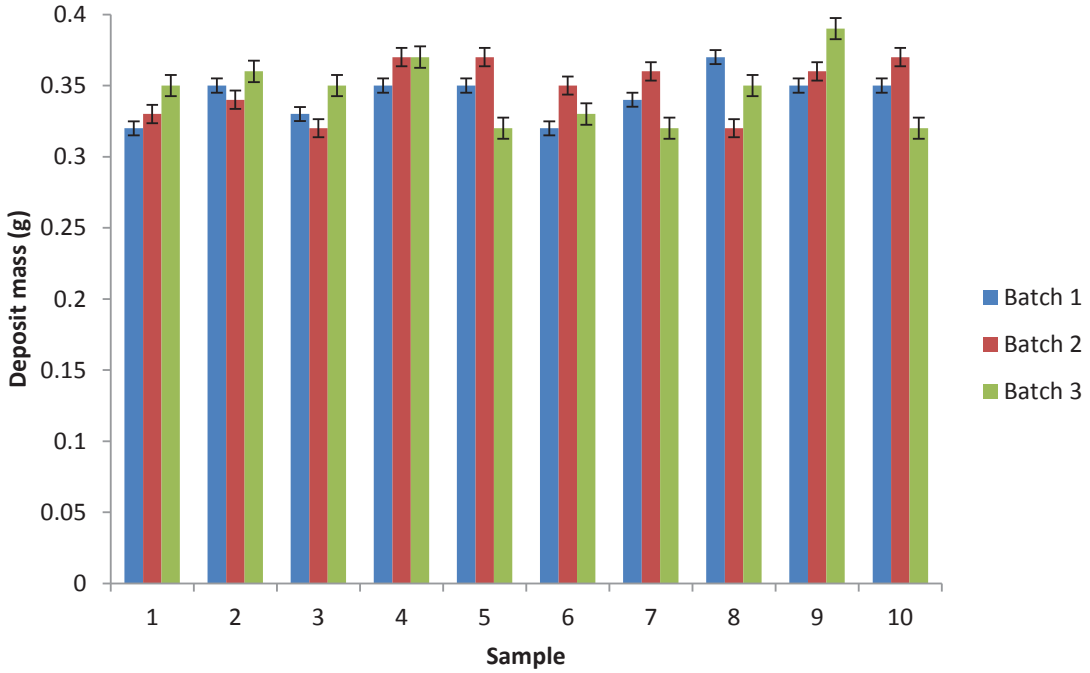


Figure 3.2 Initial deposit (a) mass and (b) thickness

3.2.2 Measurement techniques

Three parameters were set for each individual experiment, i.e. cleaning solution, stirring condition and sample configuration. All experiments were done in triplicates. After 120 minutes of treatment, the moisture content of the deposit, deposit removal performance and swelling of the deposit were calculated. The moisture content of the deposit was calculated as follows (1):

$$\textit{Moisture content (\%)} = \left(\frac{Deposit\ wet\ weight - Deposit\ dry\ weight}{Deposit\ wet\ weight}\right) X\ 100\% \qquad (1)$$

The deposit removal performance was measured by mass difference before and after experiment. In some occasions, the deposit was completely removed within 120 minutes. The weight of the coupon before fouling was measured as the empty weight. After the samples were baked, the weight of the coupon with baked deposit was measured as the initial weight. The coupon was dried out in the oven for 60 minutes at the temperature of 90℃ after the experiment completed (120 minutes), and the final weight of the coupon was measure. Thus, the removal performance could be calculated by the following equation (2):

$$\textit{Deposit removal (\%)} = \left(\frac{initial\ weight - final\ weight}{intitial\ weight - empty\ coupon\ weight}\right)\ x\ 100\% \qquad (2)$$

Image analysis (Imagej) was used to observe the swelling of egg yolk deposit when interacting with water or other cleaning solutions during hydration experiment. Images of the samples were captured from the side view at every 15 minutes before and during

the experiment. Section a, b, c, and d in the image (figure 3.3) were an equal division of the fouled surface of the coupon, while i, ii, iii, and iv represent the deposit thickness point for section a, b, c, and d respectively. An average deposit thickness of a, b, c, d can be calculated as follows; *Deposit thickness* $_{average} = \frac{i+ii+iii+iv}{4}$, which was set as the initial deposit thickness. After a certain time of experiment, the average deposit thickness can be calculated again in the same way set as the swelling deposit thickness, and thus the deposit swelling can be worked out using equation (3);

$$Deposit\ Swelling\ (\%) = \left(\frac{swelling\ deposit\ thickness - initial\ deposit\ thickness}{initial\ deposit\ thickness}\right)\ x\ 100\% \qquad (3)$$

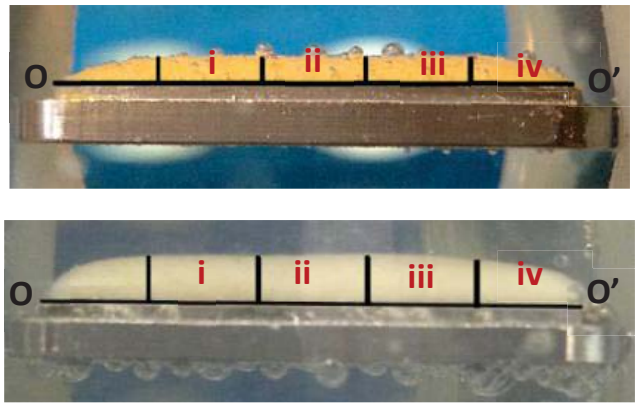


Figure 3.3: Measuring of deposit swelling using image analysis

The deposit thickness and the swelling performance can be measured using ImageJ software. First, the image taken by the USB camera was cropped using an image editing software, only the deposit and the coupon section remains in the image/photo. The deposit thickness was then measured by ImageJ software, where the length between O and O' which was already known (25 mm) has been set as a reference

scale, thus the lengths of i, ii, iii, iv (the deposit thickness) were measured automatically by the software. In Figure 3.3 the initial and swelling deposit thickness for picture (a) and (b) were 1.1 mm and 2.2 mm respectively, and the swelling performance was 100% (Table 3.1).

Table 3.1: Image analysis results by ImageJ software

	Picture (a)	Picture (b)
Length of OO' set ± 0.1(mm)	25	25
Thickness of segment a ± 0.1 (mm)	1.1	2.2
Thickness of segment b ± 0.1 (mm)	1.1	2.3
Thickness of segment c ± 0.1 (mm)	1.2	2.3
Thickness of segment d ± 0.1 (mm)	1.1	2.1
Average thickness ± 0.1 (mm)	1.1	2.2
Swelling performance	100%	

$$\text{swelling ratio} = \frac{\delta_f - \delta_0}{\delta_0} \qquad (1)$$

$$\text{swelling rate} = \frac{\delta_f - \delta_0}{t_f - t_0} \qquad (2)$$

3.2.3 Hydration experiment set-up

The coupon was held by a clamp which was fixed to a retort stand and soaked in the cleaning solution in the middle of the 2L beaker on a Stuart US152 hot plate & stirrer (Stuart, UK), which provided heating and stirring (Figure 3.4). Four types of cleaning solutions were used in this set of experiment i.e water, sodium hydroxide solution (1% w/v), LAS solution (1% w/v), and detergent solution (1% w/v) with 3 stirring conditions i.e without stirring, stirred by magnetic stirrer and stirred by mixing impeller. Samples were placed in 2 different configurations i.e horizontal and vertical configuration.

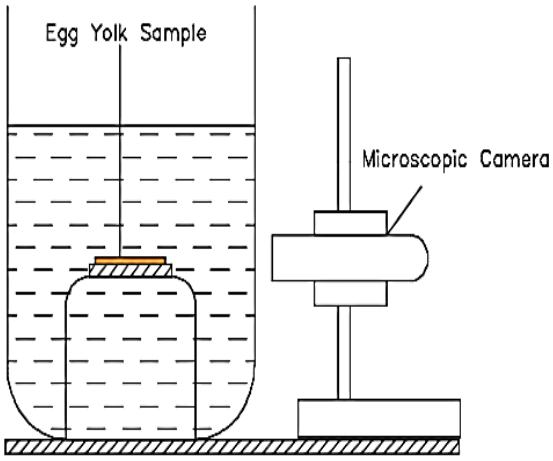


Figure 3.4: Experimental set-up for hydration experiment of egg yolk deposit

3.2.4 Hydration experiment procedure

In this study, egg yolk deposit samples were soaked in water, sodium hydroxide solution, LAS solution and detergent solution at room temperature. Apparently, the deposit samples were soaked in 2 different sample configurations i.e. vertical and horizontal with magnetic stirrer and mixing impeller used (400 rpm) to create mixing and provide shear force, and a clamp which attached to a retort stand was used to hold the samples coupon and the USB microscope camera (Veho VMS 004) throughout the hydration experiments. The sample coupon was soaked in the middle of the beaker and the beaker (2 litres) was placed on a hotplate stirrer. Initial weight of the deposit samples was measured prior to the experiments, and the initial image of the deposit samples were captured. During experiment, the image of the deposit were captured and the wet weight of the deposit samples was measured at 1, 5, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110 and 120 minutes of hydration,. It is crucial to ensure that any unsoiled surfaces of the sample coupon are dry before the wet weight of the sample was

measured. The samples were then dried in the oven at 90^{o}C for an hour. Then, the dry weight of the deposit samples was measured.

The cleaning effects of various cleaning solutions were investigated, i.e. tap water (as a control), 1% (w/w) sodium hydroxide and 1% w/w sodium hydroxide, 1% w/w LAS solution and 1% w/w commercial detergent solution. The reason using tap water rather than distilled water was for simulating industrial practice as much as possible, in which tap water was a more preferred choice. 1% w/w sodium hydroxide solution were prepared adding water into sodium hydroxide powder (Sigma Aldrich), 1% w/w LAS solution was prepared by adding water into LAS paste (Procter and Gamble), and 1% w/w detergent solution was prepared by adding water into detergent powder (Ariel). The pH values of water and all alkaline solutions were measured by a PHH-37 handheld pH meter (Omega, UK). After sticking the probe to the solution for 2 or 3 minutes, the figure on the monitor was stable. That stable figure was taken as the pH value of the solution.

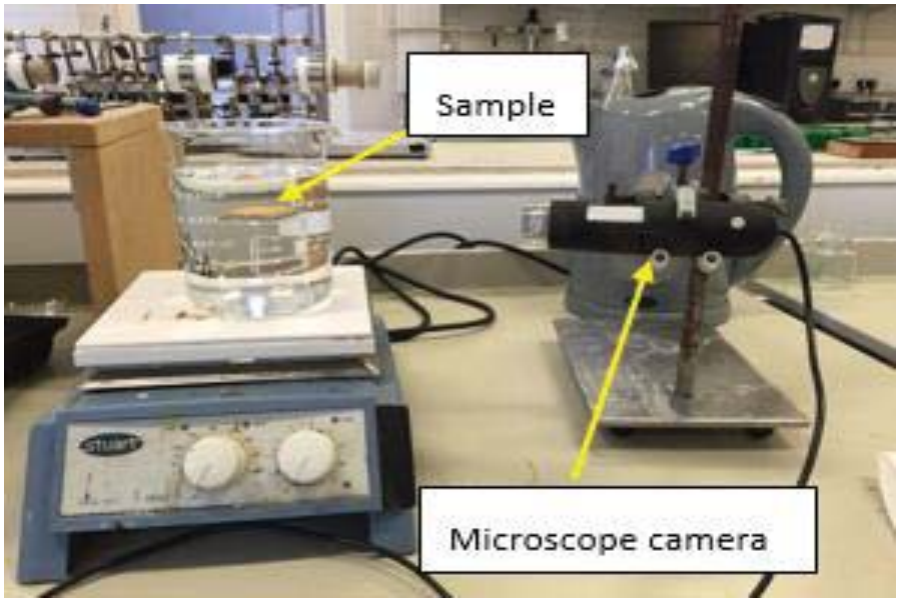


Figure 3.5: Picture of hydration experiment set-up

3.2.5 Hydration experiment via static testing

In the static system, fluid is in stationary state. There is no motion of a fluid layer to an adjacent layer. Fluid velocity is zero and there are no shear stresses. The pressure variation is due only to the weight of the fluid which is known as hydrostatic pressure. In the present studies, static testing system is used where deposit samples are soaked in cleaning fluids i.e. water, sodium hydroxide solution, LAS solution, and commercial detergent solution are placed in a 2 liter beaker without stirring.

3.2.6 Hydration experiment via dynamic testing

In the dynamic system, there is motion of a fluid layer to an adjacent layer and the fluid velocity creates shear stress on a surface. Surface shear stress is influenced by the flow rate of the fluid which means an increase flow rate results in higher surface shear stress. In the present study, dynamic testing system is designed where deposit samples soaked in cleaning fluids i.e. water, sodium hydroxide solution, LAS solution, and commercial detergent solution filled in a 2 liter beaker and stirred using magnetic stirrer bar and a pitched blade turbine impeller at 400 rpm to create shear stress on the deposit samples.

3.2.7 Flow patterns in hydration experiment

Magnetic stirrer and pitch blade turbine impeller were used in this study to create fluid flow at 400 rpm. Basically, this magnetic stirrer comprises of a rotating magnet with adjustable frequency around a fixed vertical axis below a flat horizontal surface. The rotation of this magnet rotates the magnetic stirrer bar on the bottom the beaker (2 liter) placed on the magnetic stirrer (Halasz et al., 2008). The rotation of the magnetic stirrer

bar creates a rotational flow of the liquid filled in the vessel (cleaning fluid) by spinning the fluid around to create a vortex. This vortex flow helps to break the surface tension of the liquid which is useful for mixing. It is well understood that the rotation of pitched blade turbine impeller creates axial flow in a vessel filled by liquid (Bakker et al., 1996). This impeller use blades angled to the vertical with no disk is present. Each blade is angled downward so that when blade rotates, the liquid flow moves down towards the bottom of the vessel, and when the fluid flow hits the bottom of the vessel, the fluid bounces off and moves towards the sides of the vessel (left and right). The extension force of the spinning blade pushes the liquid flow upwards and starts to recirculate when the flow as the rotating blade pulls the liquid from above.

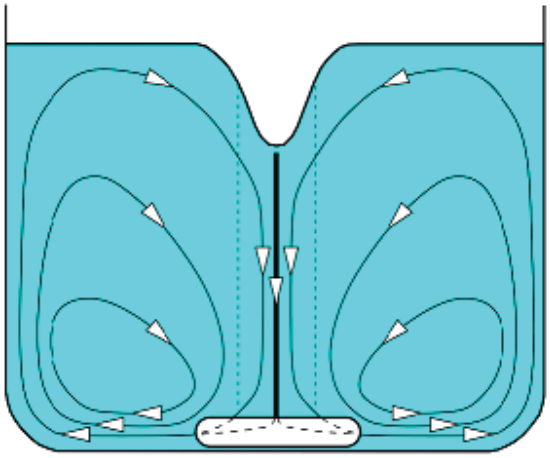


Figure 3.6: The flow pattern of fluid generated by a magnetic stirrer bar (Halasz et al., 2008)

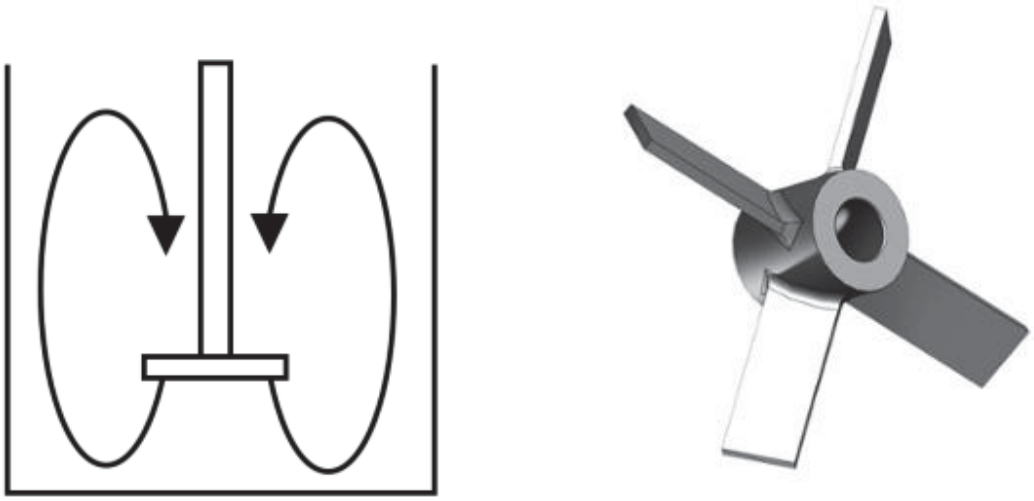


Figure 3.7: a) The flow pattern fluid generated by pitched blade turbine impeller b) Picture of pithed blade turbine

3.2.8 Cleaning rig set-up

The cleaning rig used to study the cleaning of egg yolk deposits was previously known as flow cell rig or coupon rig (Christian, 2004; Aziz, 2007; Goode, 2012; Cole, 2013). This cleaning rig consists of a stainless steel rectangular flow channel (7 mm height and 32 mm height) which contains a test section located in the middle of the flow channel, a microfoil heat flux sensor (MHFS) unit, a chemical tank, a heating tank, a centrifugal pump, flow transmitter PD340, thermocouples, valves, a data logger and a computer.

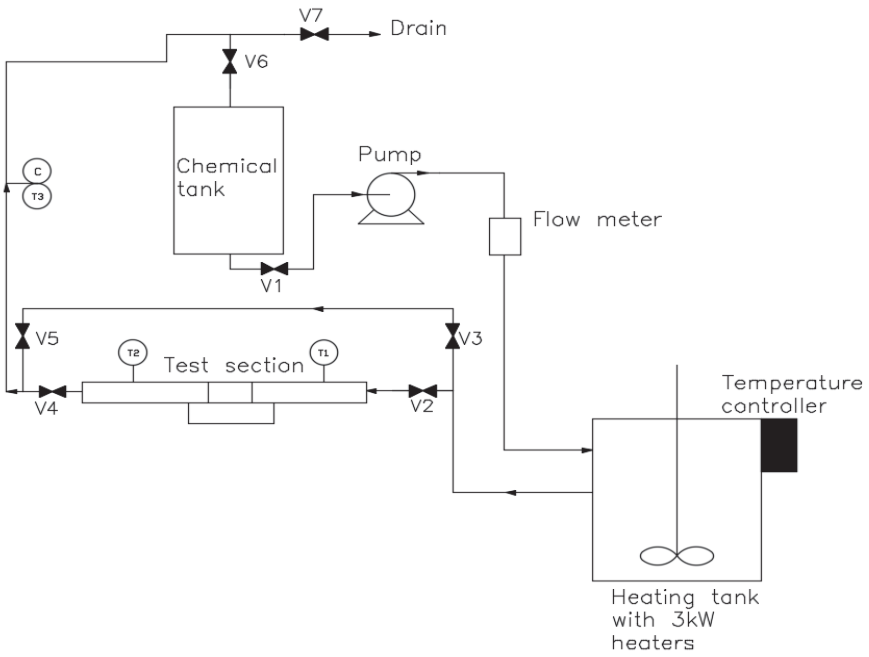


Figure 3.8: Schematic diagram of the flat plate flow cell rig by (Palabiyik et al., 2014) and (Christian and Fryer, 2006)

The fouled coupon was located in the test section which was designed with stainless steel base and glass on the side and the top of the test section to allow monitoring by camera. A thin layer of silicone grease was applied to the outer rim of the coupon to hold it on the test section base. The MHFS unit was placed in the ice box to provide a maximum temperature difference between the heat flux sensor and the cleaning fluid. Microscopic cameras (Veho VMS-004) were used to monitor the cleaning of egg yolk deposits and to capture images from side and top view for image data analysis.

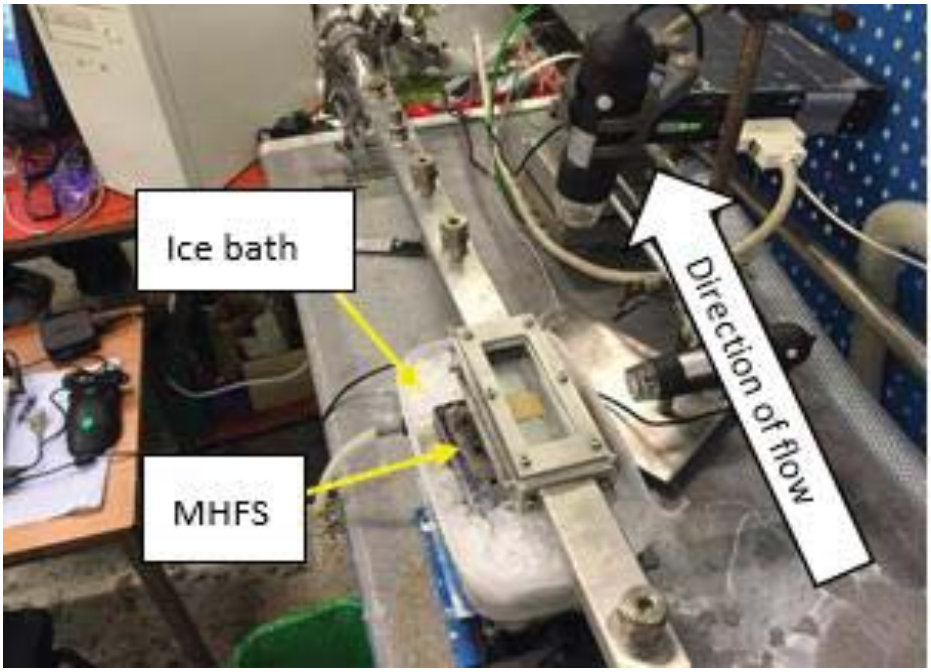


Figure 3.9: The test section set-up

There were two different parameters used to monitor the cleaning i.e.:

i) Heat transfer coefficient (U) calculated from the measured heat flux (q), the average temperature of the test section cleaning fluid (T_{av}), and the temperature of the insulated thermocouple placed below the fouled coupon (T_{c2}) as described in section 2.15.

ii) The determination of area of deposit removed (mm^2) via image analysis (imageJ) which will be described in section 3.34.

All experiments were conducted at 0.5 ms^{-1} flow rate. Three different temperatures (40^oC, 60^oC and 80^oC) were used in this set of experiments with three different cleaning agents were applied (water, sodium hydroxide solution and LAS solution) at two different chemical concentrations (1 % and 5 % w/v). All experiments were carried out in triplicates.

3.2.9 Cleaning rig experiment procedures

The initial deposit weight was measured and the cleaning fluid was heated up prior to the experiment by circulating it from the chemical tank throughout the heating tank and then goes back to the chemical tank. At this stage, the test section valves were set in a condition that the cleaning fluid is not allowed to enter the test section. The cleaning fluid was let to circulate until it reaches the desired temperature. Then, the test section valves were opened to allow the cleaning fluid passes through the test section. The experiment starts off when the cleaning fluid enters the test section and then, the data logger was set to start recording the data and the cameras start to capture image every 5 minutes. The experiment was stopped when the deposit was totally removed (visually monitored) or if the deposit was not completely removed after 4 hours of experiment. Then, the sample coupon was taken out and the weight of deposit left was measured. The cleaning fluid was flushed out through the drain and the whole rig was flushed with water in order to ensure that no chemical or deposit fractures left in the system.

3.2.10 Image analysis for deposit removal

Deposit removal at every 5 minutes of experiment was determined using images captured which were analyzed using ImageJ. A reference scale needs to be set up before the measurement was carried out. This reference scale was set up based on a known distance, for example the width of the coupon (figure 3.10). Then a line needs to be drawn to mark the deposit left area needed to be analyzed and the area (mm^2) of the deposit left will be measured by the software (figure 3.11). The deposit removal was determined from the area of the deposit left on the coupon as follows;

$$\text{The deposit removal (\%)} = \frac{625\ \text{mm}^2 - \text{area of deposit left}}{625\ \text{mm}^2} \times 100\% \qquad (4)$$

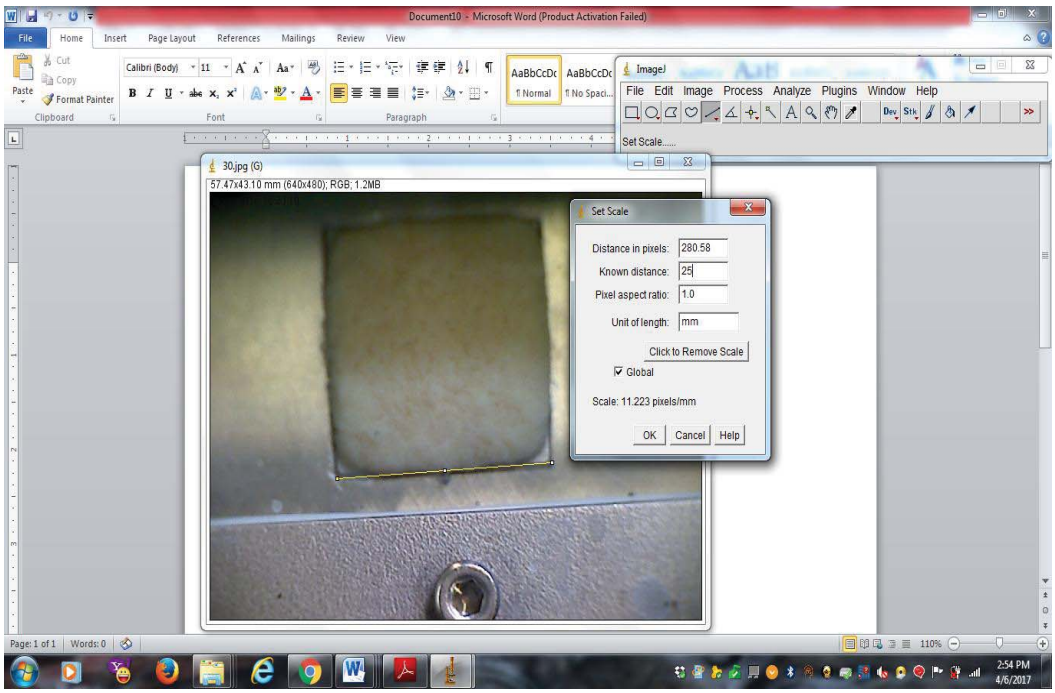


Figure 3.10: Setting up a reference scale for area measurement

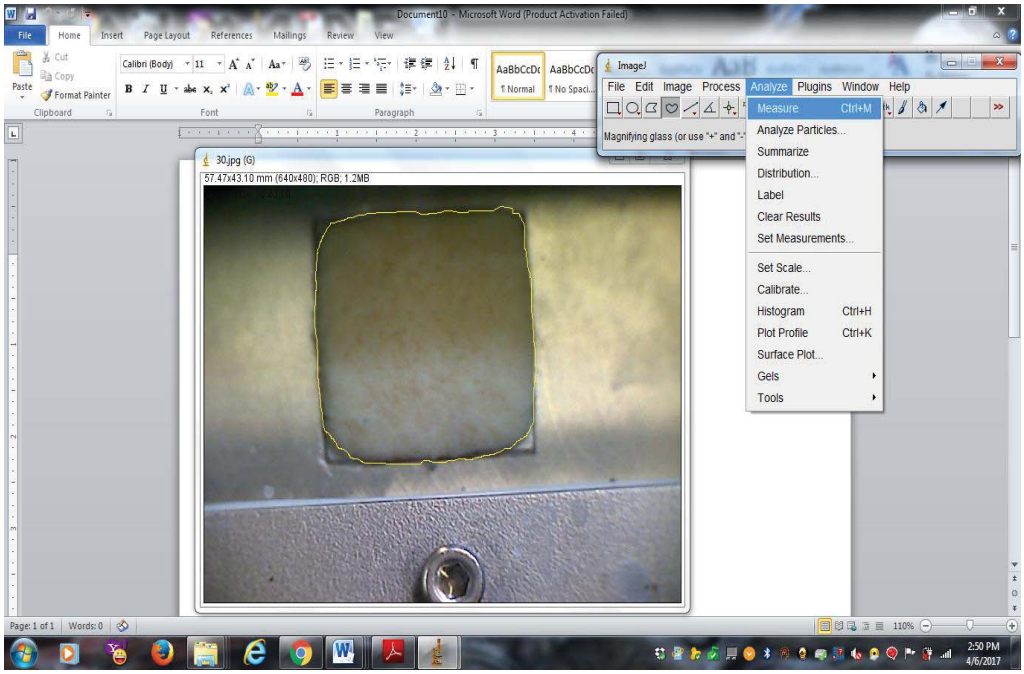


Figure 3.11: Measuring the area of deposit left using ImageJ

3.2.11 Micromanipulation rig operation procedure

Set of experiments were conducted in which the egg yolk deposits were fouled on to rectangular coupons (25 mm x 25 mm) and presoaked in water, sodium hydroxide solution, and LAS solution at different hydration time (15 and 30 minutes), hydration temperature (20, 40 and 60^{o}C) and chemical concentrations (1 and 5 % w/v) prior to measurement by micromanipulator. Then, shear force was applied on the samples using micromanipulator to measure the force required to remove the egg yolk fouling deposits, in terms of adhesive and cohesive strength. A T-shaped probe which connected to a force transducer (Model BG-1000, Kulite semiconductor, Leonia, NJ, USA) was used to provide shear force on the deposits sample (Liu *et al*., 2007). The coupon containing the egg yolk deposits was then placed on a stage that attached to a micromanipulator (MicroInstruments, Oxon, UK). This micromanipulator holds microscope cameras which connected to the monitors and a computer was used to

control the operation. The gap between the probe and the coupon surface was adjusted to 10 μm through a side-view monitor. The width of the probe was made larger than the coupon width to minimize edge effects (Liu *et al*., 2007). The measurement was carried out using the probe to pull the deposits away from the surface horizontally, at a constant speed of 1.11mm/s. A multifunctional data acquisition board (Amplicon Liveline, Brighton, UK) was used to record the force exerted on the probe at 100 Hz (Liu *et al*., 2007). Data collected from the experiments were analyzed according to the calculation shown in section 2.13. All experiments were carried out in triplicates.

The force transducer used in the measurement needs to be calibrated prior to measurement in order to ensure that the data collected are accurate. Calibration was conducted using standard weights (50 g) provided (figure 3.12) and graph for voltage output (V) versus mass (Kg) was plotted (figure 3.13). Calibration factor obtained from the graph needs to be used in the calculation to determine the adhesive and cohesive strength of deposits.

In cohesive strength measurement, the gap between the probe and the coupon surface was adjusted to 1 mm. This is to ensure that the probe pull the top layer of the deposit away from the coupon and leaving the bottom layer on the coupon surface to break the cohesive bond between the deposits.

Figure 3.12: Standard weights used in the calibration of the force transducer

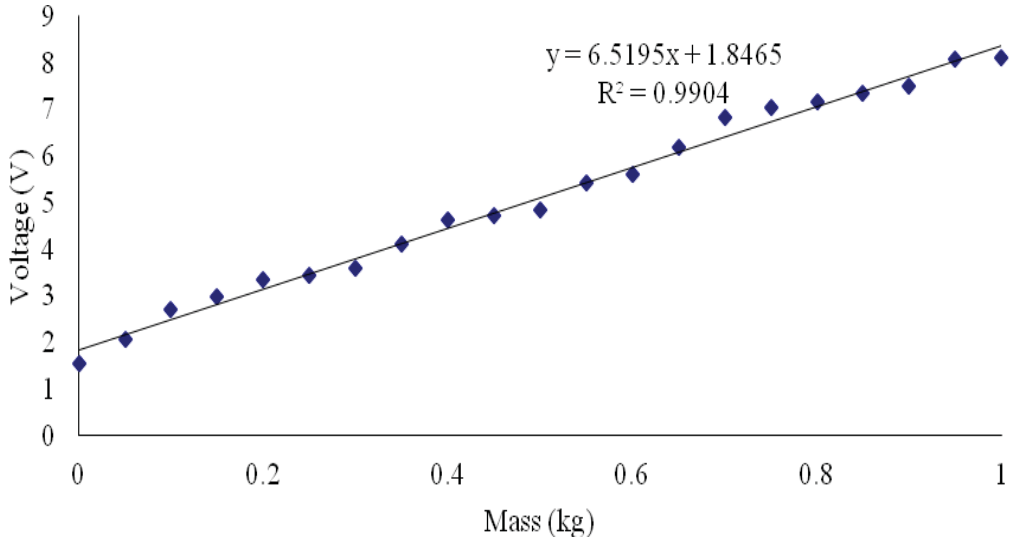


Figure 3.13: Calibration curve for 1000 g transducer (Rois Anwar, 2016)

3.2.12 Statistical Analysis

All experiments were carried out in triplicates for all parameter tested. Standard error was obtained using analysis of variance (ANOVA) in Microsoft excel. Statistical analysis was carried out using the student t-test in Microsoft Excel where differences were considered significant at $p<0.05$.

CHAPTER 4: THE EFFECT OF VARIOUS PARAMETERS ON SWELLING AND REMOVAL OF EGG YOLK DEPOSITS DURING HYDRATION

4.1 Results

4.1.1 Effect of different cleaning solutions on the moisture content of egg yolk deposits

Sets of experiments on hydration of egg yolk deposits were carried out to study the effect of different cleaning agent on the moisture content of the egg yolk deposits. In these experiments, egg yolk deposit samples were soaked in 4 different cleaning agent i.e. water, sodium hydroxide solution (1% w/w), LAS solution (1% w/w) and commercial detergent solution (1% w/w) at 20^{o}C at horizontal sample configuration with stirring at 400 rpm (magnetic stirrer). All experiments were carried out in triplicates. Figure 4.1 shows that the samples soaked in 1% w/w sodium hydroxide solution gives the highest moisture content (61.9% $\pm$ 1%) after 120 minutes of hydration followed by the samples soaked in 1% w/w LAS solution (54.39% $\pm$ 1%), the samples soaked in 1% detergent (49.55% $\pm$ 1%), and the samples soaked in water (38.57% $\pm$ 1%). At 1 minute hydration, there is only a slight significant difference in moisture content was shown between samples soaked in water and 1% w/w LAS solution, and 1% w/w detergent solution (23.53% and 22.2% $\pm$ 1%). Experiments also suggest that samples soaked in water has the lowest hydration ability compare to samples soaked in 1% w/w sodium hydroxide solution, 1% w/w LAS solution and 1% w/w detergent solution after 120 minutes of hydration.

Water-protein interaction is an important factor in the dehydration, rehydration, solubility, viscosity, gelation, heat stability, browning reaction, and other important

properties of food products over a wide range of water activities. This phenomenon is a function of pH, temperature, protein concentration, total solids concentration, and ionic composition (Morr, 1989). It has been stated in the literature that not every detergent is able to stabilize the native structure of a protein (Bowie, 2001). Additionally, detergents affect the kinetics of the reconstitution process because of their critical micellar concentration (CMC) and association constants with different lipids and proteins (Kaufmann, 2006). An excess amount of detergent can cause loss of quaternary and tertiary structure and protein aggregation.

Moreover, an excess of detergent slows down the kinetics of detergent removal. The nature and amount of ions present in the hydrating solution may also steer the interactions of proteins with each other. The possibility of counter-ions to interact with charged residues on proteins as well as lipids (head group) governs the electrostatics of intermolecular association. Divalent cations are mainly acting on the intra-structural level of a protein providing stability to charged regions. Furthermore, ions affect the water structure, thereby implicating thermodynamical aspects which leads to increase in the salt concentration in a protein solution which causes changes to the hydration of protein, because the ions themselves need water for solvation; decrease of repulsive electrostatic protein-protein interactions which allows the molecules to come closer; and direct interaction of ions with residues at the surface of protein to form a protein salt (Kaufmann, 2006).

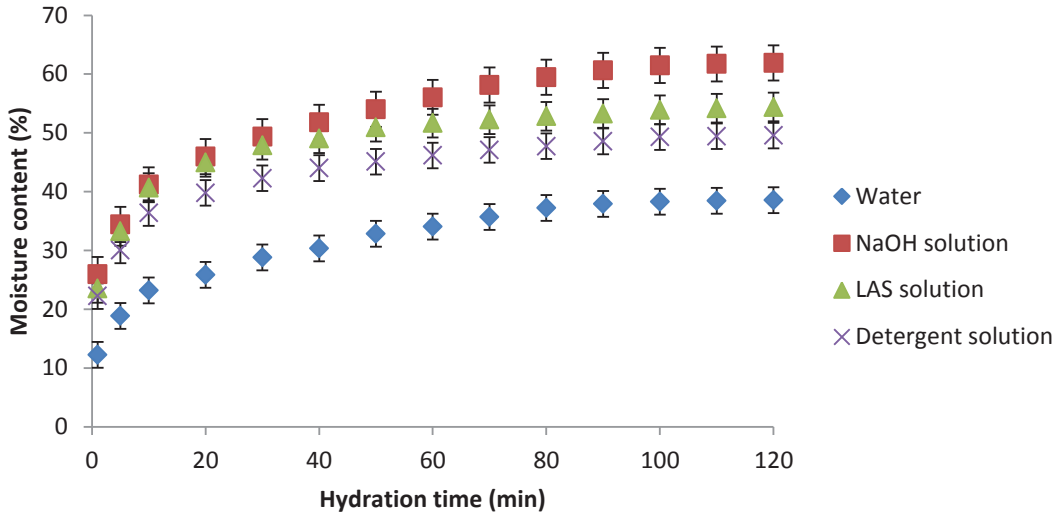


Figure 4.1: Moisture content of egg yolk deposits soaked in water, 1% w/w, sodium hydroxide solution, 1% w/w LAS solution and 1% w/w detergent solution at 20°C at horizontal sample configuration using 400 rpm stirring using magnetic stirrer

4.1.2 Effect of different hydration temperature on the moisture content of the deposit

In these sets of experiments, the effect of different hydration temperature was investigated with egg yolk deposit samples were soaked in water at three different temperatures at horizontal sample configuration using magnetic stirrer at 400 rpm. All experiments were carried out in triplicates. Figure 4.2 shows that samples soaked in water at 80°C have the highest moisture content (62.39 ± 1%) after 120 minutes of hydration in water, followed by samples soaked in water at 60 °C (56.87 ± 1%) and samples soaked in water at 40°C (50.12 ± 1%). All samples are going through the similar trend with rapid increase in moisture content during the first 60 minutes of experiment and the moisture content increment starts to reduce gradually after 80 minutes. Kaufmann (2006) suggested that the temperature of a hydration solution

affects the lipid phase and the protein stability. Additionally, it acts on the kinetics of the experiments. At higher temperature, the diffusion controlled removal of deposits is faster than lower temperature.

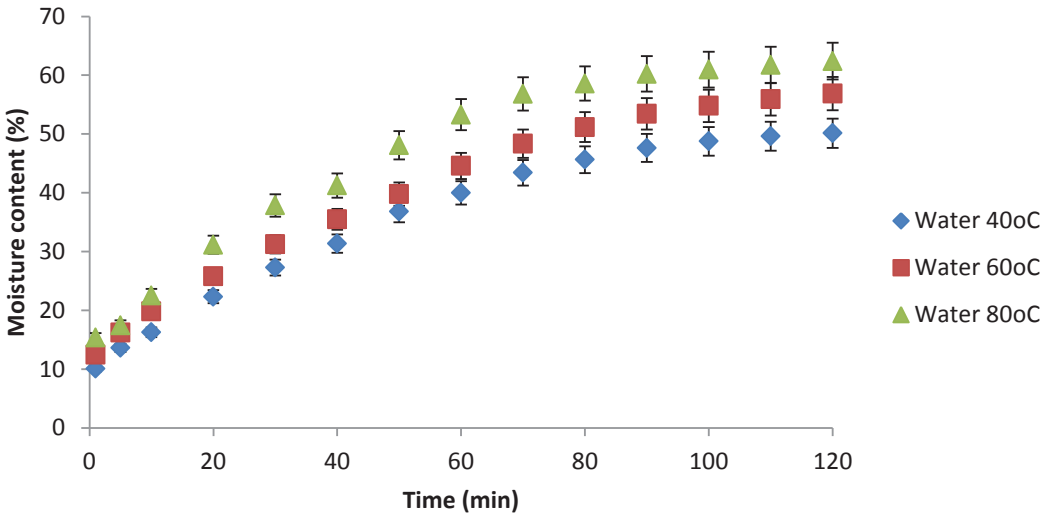


Figure 4.2: Moisture content of egg yolk deposits soaked in water at 40, 60 and 80 in horizontal sample configuration and with 400 rpm stirring using magnetic stirrer

4.1.3 Effect of different stirring speeds on moisture content of egg yolk deposit

A series of experiments were carried out to study the effect of different stirring speed on moisture content of egg yolk deposits. Samples were soaked in water at 20^{o}C at 3 different rotational speed i.e 0, 200 and 400 rpm using mixing impeller. All experiments were carried out in triplicates. At 1 minute of hydration, figure 4.3 shows that samples soaked in water at 200 rpm and 0 rpm show quite similar moisture content (12.2 $\pm$ 1% and 12.25 $\pm$ 1%) while samples soaked in water at 400 rpm show higher moisture content (15.78 $\pm$ 1%). After 120 minutes of experiments, samples soaked in water at

400 rpm give the highest moisture content (42.19 ± 1%) followed by samples soaked in water at 200 rpm (38.57 + 1%) and samples soaked in water without stirring (33.33 ± 1%).

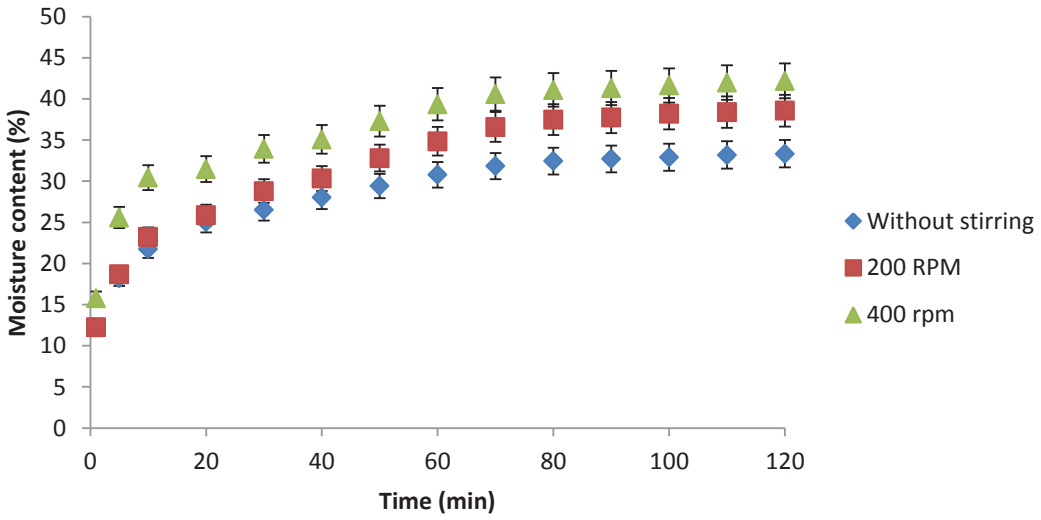


Figure 4.3: Moisture content of egg yolk deposit samples soaked in water at different stirring speeds at 20°C at horizontal sample configuration.

4.1.4 Effect of different sample configurations on moisture content of egg yolk deposit

In these sets of experiments, the effect of different sample configurations was studied. Egg yolk deposit samples were soaked in water and 1% w/w sodium hydroxide solution, 1% w/w LAS solution and 1% detergent solution in 2 different samples configuration i.e horizontal and vertical at 20°C and 400 rpm using magnetic stirrer. All experiments were carried out in triplicates. Figure 4.4(a) shows that samples soaked vertically in 1% sodium hydroxide solution have the highest moisture content (66.96 ± 1%) after 120 minutes of hydration followed by samples soaked horizontally in 1% sodium hydroxide solution (61.90 ± 1%), samples soaked vertically in water (41.54 ± 1%), and samples

soaked horizontally in water (38.57 ± 1%). There is only a slight significant difference in moisture content between samples soaked vertically and horizontally in both hydrating solution after 120 minutes of hydration. Figure 4.1.4(b) shows that samples soaked vertically in 1% LAS solution gives the highest moisture content (60.27 ± 1%) after 120 minutes of hydration followed by samples soaked horizontally in 1% w/w LAS solution (54.39 ± 1%), samples soaked vertically in 1% detergent (56.32 ± 1%), and samples soaked horizontally in 1% w/w detergent (49.55 ± 1%). There is only a slight significant difference in moisture content between samples soaked vertically and horizontally in both 1% w/w detergent solutions after 120 minutes of hydration, while samples soaked vertically and horizontally in 1% w/w LAS solution shows a significant difference in moisture content. Experiments also indicate that samples soaked in vertical position have higher hydration ability compare to samples soaked in vertical position after 120 minutes of hydration.

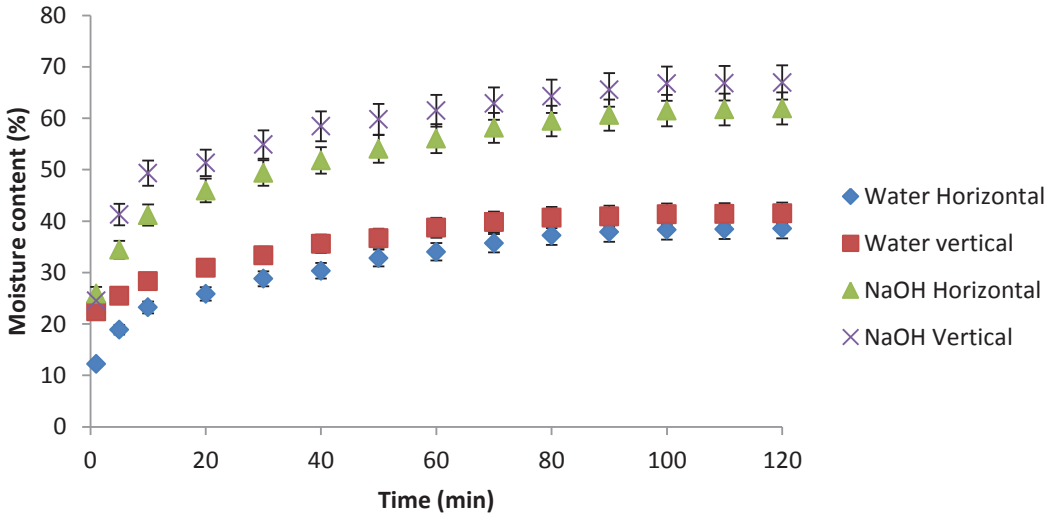


Figure 4.4(a): Moisture content of egg yolk deposit sample soaked in water and 1% w/w sodium hydroxide solution at 20^{o}C and 400 rpm with magnetic stirrer in horizontal and vertical sample configuration.

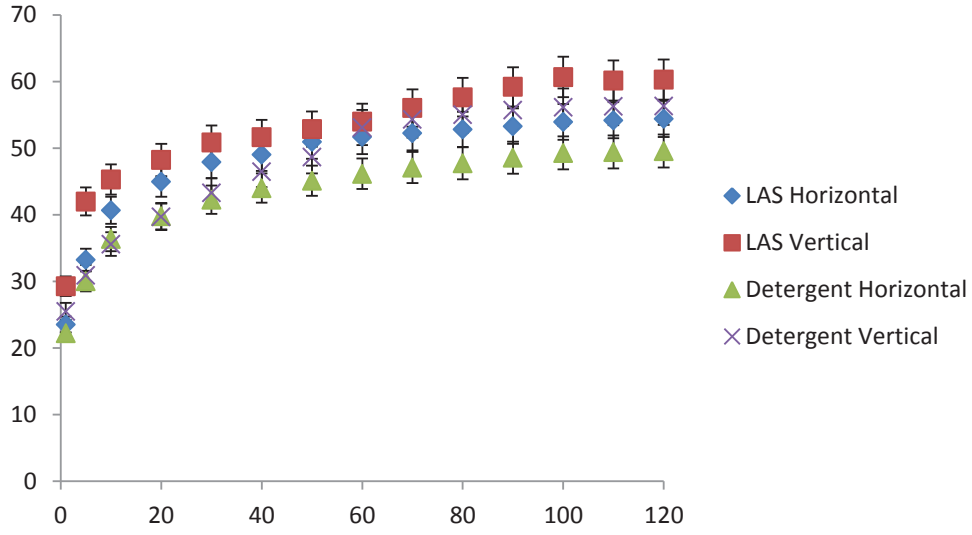


Figure 4.4 (b): Moisture content of egg yolk deposit sample soaked in 1% w/w LAS solution and 1% w/w detergent solution at 20°C and 400 rpm with magnetic stirrer at horizontal and vertical sample configuration.

4.1.5 Swelling of egg yolk deposit soaked in water and sodium hydroxide solution

In this section, sets of experiments were carried out to the swelling performance of egg yolk deposits soaked in water and sodium hydroxide solution. All experiments were carried out in triplicates. The swelling performance of egg yolk deposits soaked in contact with water at three different temperatures (40, 60, 80 ℃) for non-stirring was shown in Figure 4.5 (a). Trend lines could generally fit the Type (a) swelling mode as described in the literature review section.

Figure 4.21 (b-f) shows the swelling performance of egg yolk deposits soaked in water at 0.5% w/w sodium hydroxide solution. The results for non-stirring at the three temperatures were shown in Figure 4.5 (b). Samples soaked in 0.5% w/w sodium hydroxide solution at 60°C (152 $\pm$ 5%) give the highest swelling performance followed

by samples soaked at 40^{o}C (149.18 ± 5%) and 80^{o}C (115.16 ± 5%). Samples soaked at 80^{o}C shows the lowest swelling performance because of the removal of the deposit. Results for the samples soaked in sodium hydroxide at stirring speed of 200 rpm at 40 and 60℃ were shown in Figure 4.5 (c). After 120 minutes of experiments, samples soaked at 40^{o}C (159.29 ± 5%) shows higher swelling performance than samples soaked at 60^{o}C (146.95 ± 5%) obviously because of deposit removal of samples soaked in sodium hydroxide solution at 60^{o}C were higher than samples soaked in sodium hydroxide solution at 40^{o}C. Results for samples soaked in sodium hydroxide at 200 rpm stirring speed at 80℃ were presented in Figure 4.5 (d). It shows that the swelling performance of the samples decrease because of deposit removal occurs during experiments. Results for samples soaked in sodium hydroxide solution at 400 rpm stirring were shown in Figure 4.5 (e) and Figure 4.5 (f). At 80℃, samples soaked in sodium hydroxide solution at 200 rpm and 400rpm reach 100% deposit removal after 83 and 67 minutes of experiments respectively. Trend lines for samples soaked in sodium hydroxide at 40 and 60℃ could generally fit the Type (b) swelling mode described earlier in section 2.18. In picture (d), the trend lines experienced increase, plateau and decrease stages, which demonstrated the process of swelling, plateau and erosion. Though the thickness did not represent removal, the trend would still be similar when plotting removal against time.

Figure 4.5 (g- l) shows the swelling performance of egg yolk deposit samples soaked in 1% w/w sodium hydroxide solution. The results for samples soaked in solution without stirring at 40 and 60℃ were shown in Figure 4.5(g), while results for non-stirring, 80℃ were shown in Figure 4.5 (h). Samples soaked in sodium hydroxide solution at 40^{o}C

(201.82 ± 5%) give the highest swelling performance after 120 minutes of experiments followed by samples soaked at 60°C (119.01 ± 5%) and 80°C (9.14 ± 1%). Results for samples soaked in sodium hydroxide at stirring speed of 200 rpm at 40 and 60℃ were shown in Figure 4.5 (i) and results for samples soaked in solution at 80℃ were presented in picture Figure 4.5 (j). Samples soaked in sodium hydroxide solution at 40°C (200.82 ± 5%) show the higher swelling performance after 120 minutes of experiments than samples soaked at 60°C (1.01 ± 0.05%). Results for samples soaked in solution at 400 rpm stirring at 40 and 60℃ were shown in Figure 4.5 (k) and results for samples soaked in solution at 400 rpm stirring at 80℃ were presented in Figure 4.5 (l). Samples soaked in sodium hydroxide solution at 40°C (171.5 ± 5%) show the higher swelling performance after 120 minutes of experiments than samples soaked at 60°C (61 ± 5%). The deposits were 100% removed at between 59 and 70 minutes for all samples soaked in sodium hydroxide at 80°C.

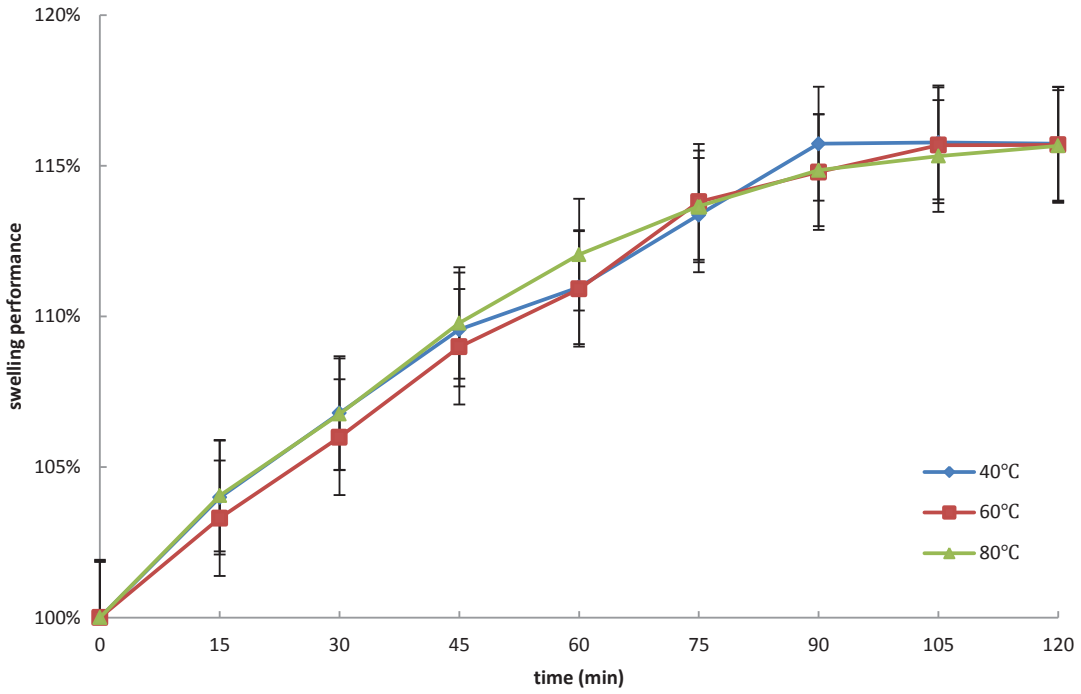


Figure 4.5 (a): Swelling performance of egg yolk deposit soaked in water at three different temperatures without stirring

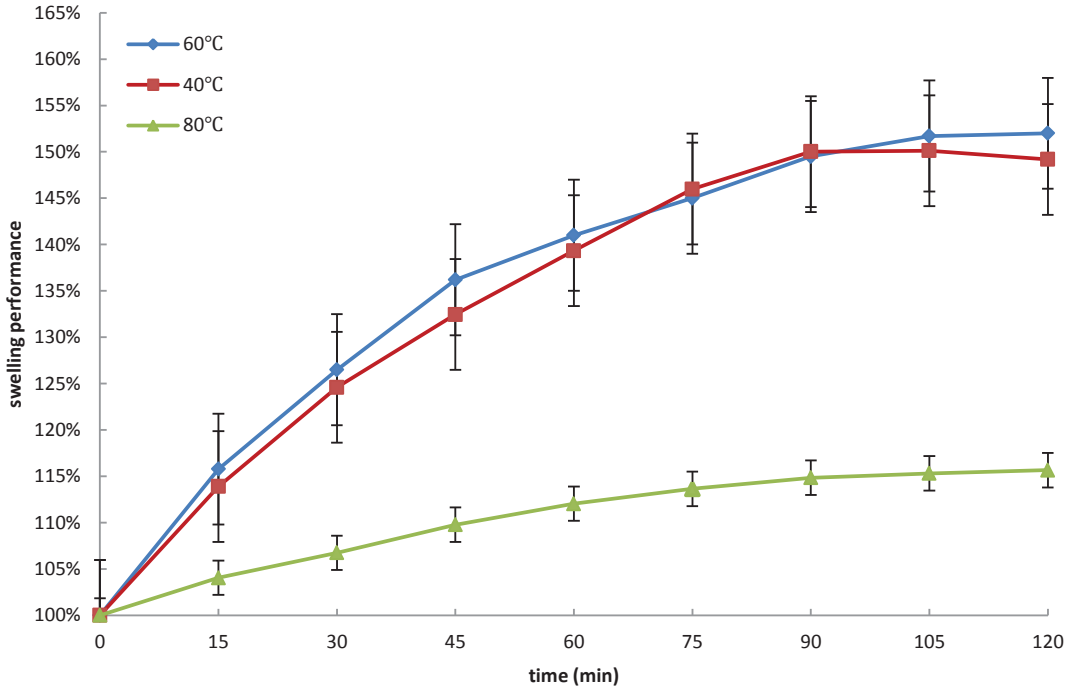


Figure 4.5(b): Swelling of egg yolk deposits soaked in 0.5% w/w sodium hydroxide solution at 3 different temperatures without stirring

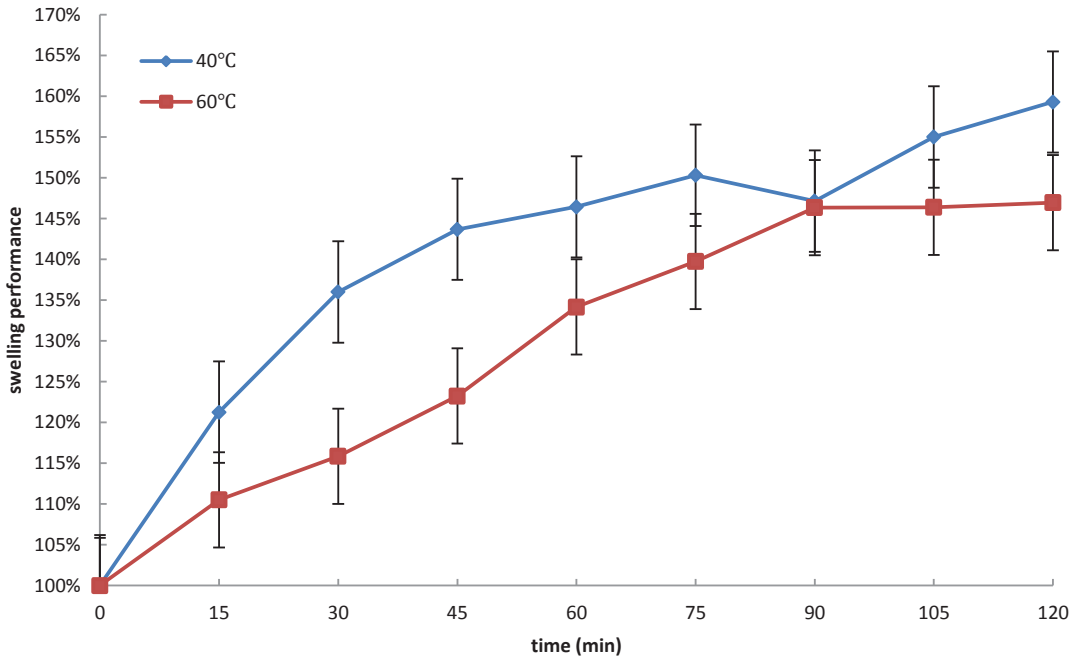


Figure 4.5(c): Swelling of egg yolk deposits soaked in 0.5% w/w sodium hydroxide solution at 40 and 60^{o}C with 200 rpm stirring

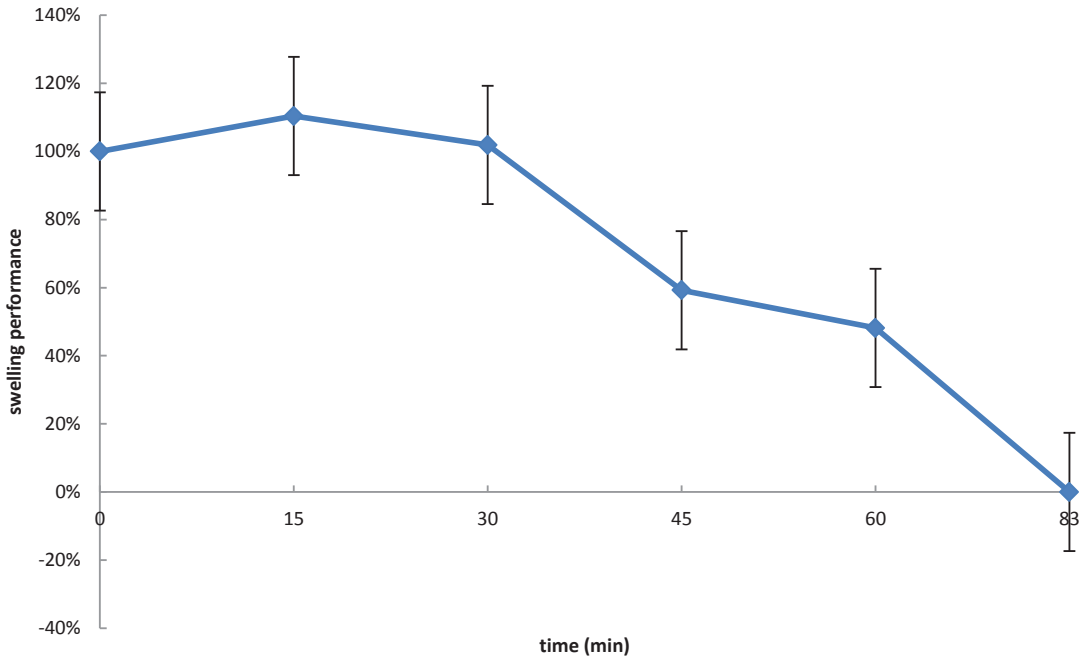


Figure 4.5(d): Swelling of egg yolk deposits soaked in 0.5% w/w sodium hydroxide solution at 80°C with 200 rpm stirring

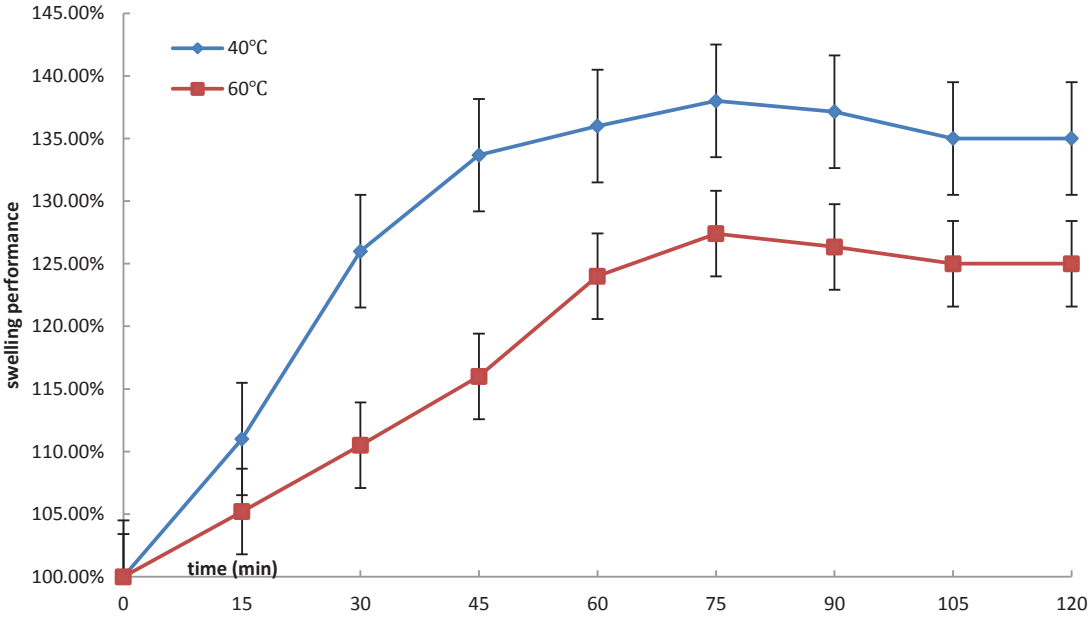


Figure 4.5(e): Swelling of egg yolk deposits soaked in 0.5% w/w sodium hydroxide solution at 40 and 60°C with 400 rpm stirring

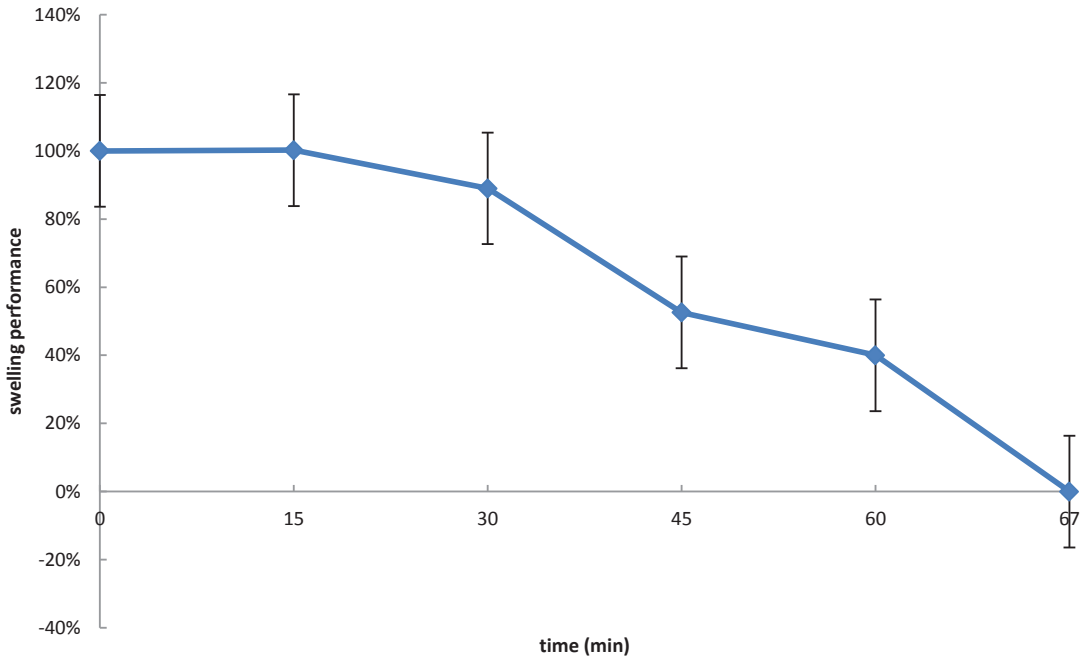


Figure 4.5(f): Swelling of egg yolk deposits soaked in 0.5% w/w sodium hydroxide solution at 80^{o}C with 400 rpm stirring

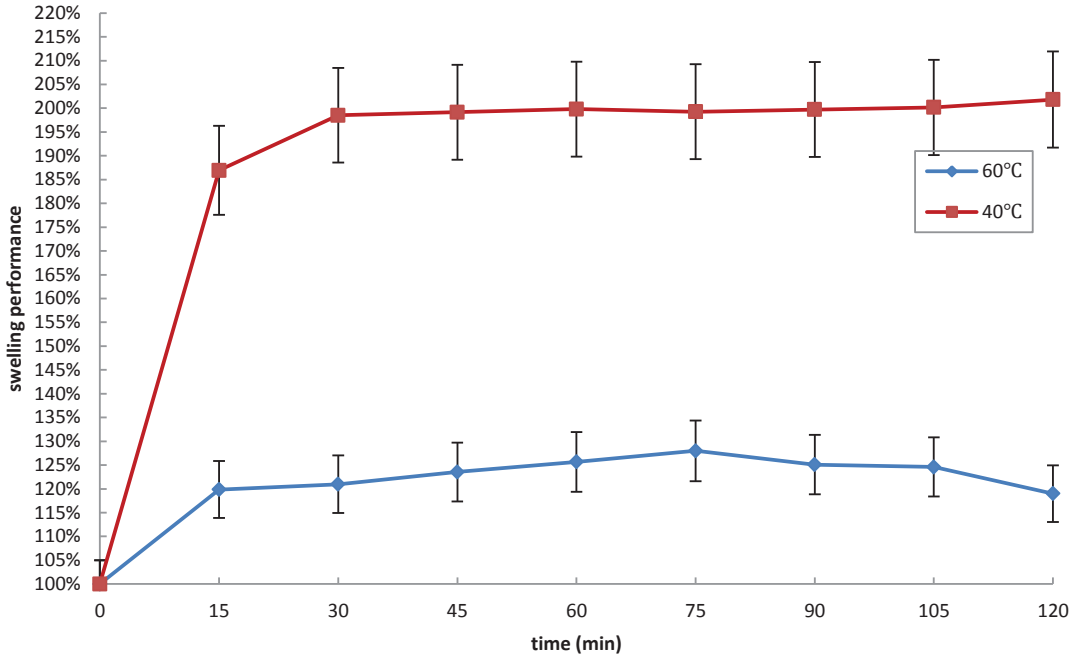


Figure 4.5(g): Swelling of egg yolk deposits soaked in 1% w/w sodium hydroxide solution at 40 and 60^{o}C without stirring

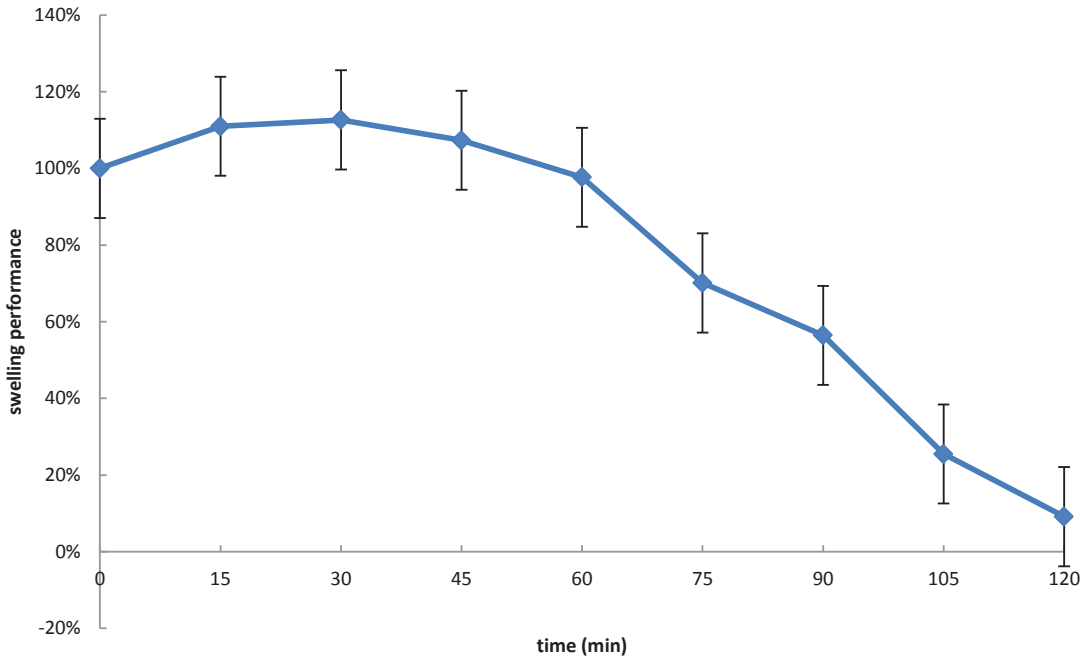


Figure 4.5(h): Swelling of egg yolk deposits soaked in 1% w/w sodium hydroxide solution at 80°C without stirring

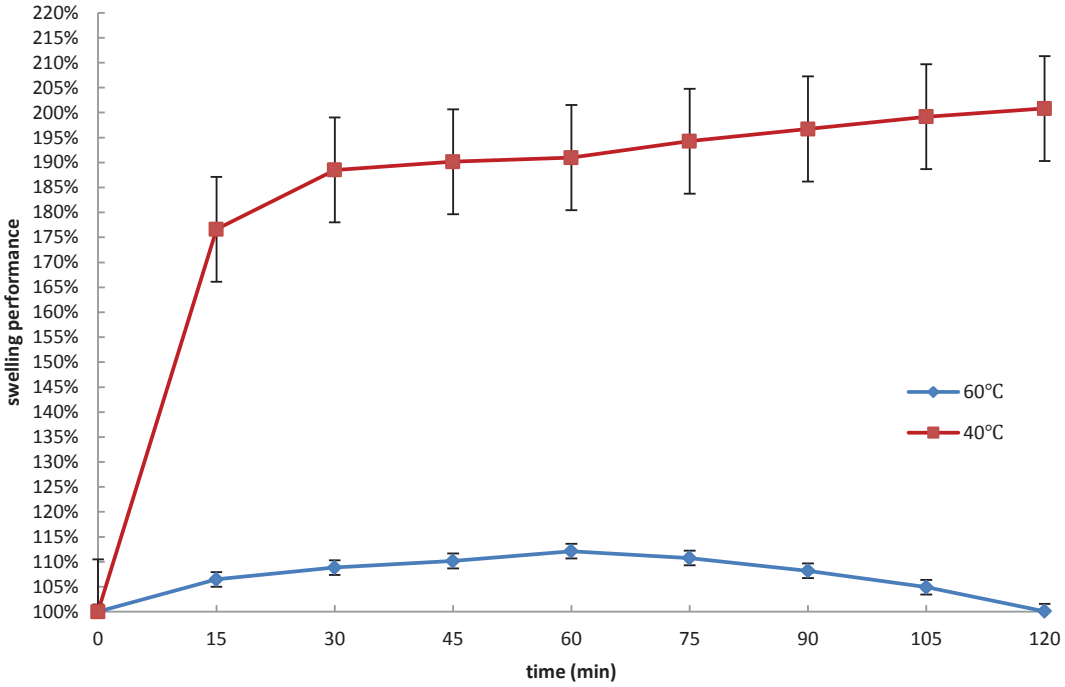


Figure 4.5(i): Swelling of egg yolk deposits soaked in 1% w/w sodium hydroxide solution at 40 and 60°C with 200 rpm stirring

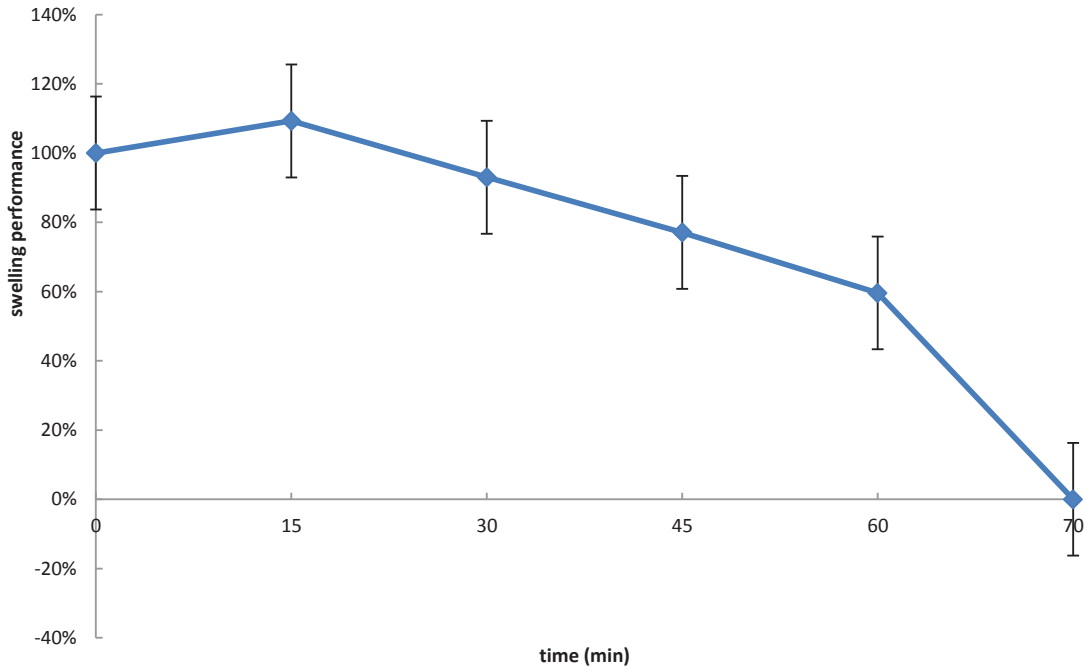


Figure 4.5(j): Swelling of egg yolk deposits soaked in 1% w/w sodium hydroxide solution at 80^{o}C with 200 rpm stirring

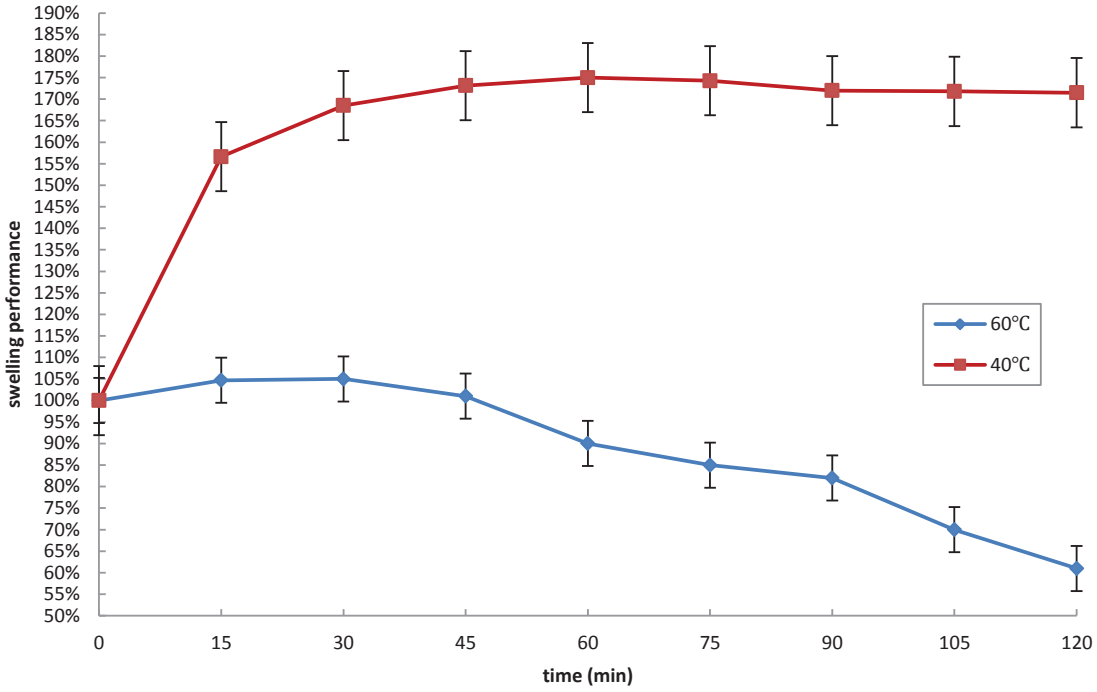


Figure 4.5(k): Swelling of egg yolk deposits soaked in 1% w/w sodium hydroxide solution at 40 and 60°C with 400 rpm stirring

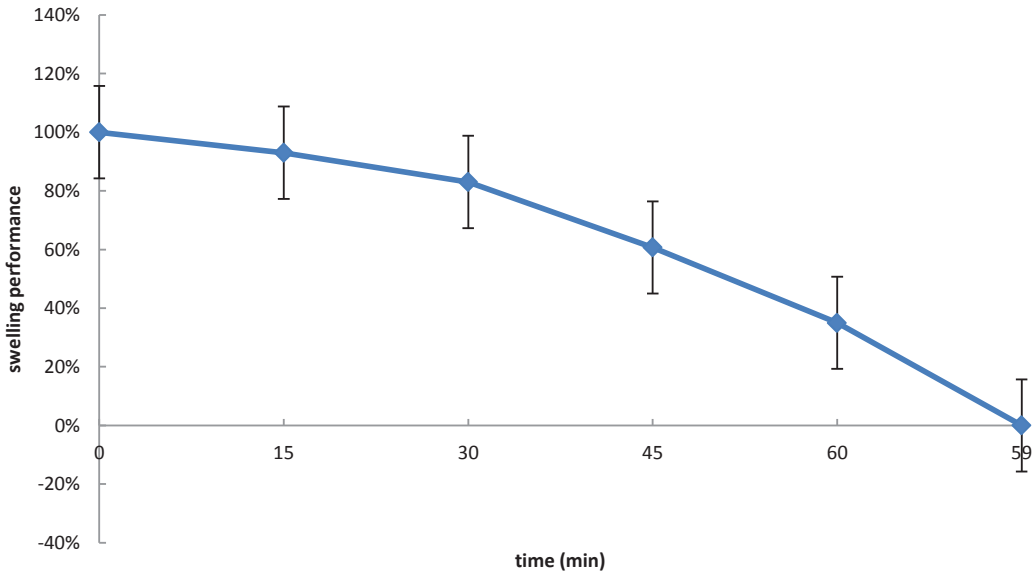


Figure 4.5(l): Swelling of egg yolk deposits soaked in 1% w/w sodium hydroxide solution at 80°C with 400 rpm stirring

4.1.6 Removal of egg yolk deposits soaked in water at different temperatures and stirring speeds.

In this series of experiments, the removal of egg yolk deposits soaked in water is studied. Samples were soaked in water at various temperatures with different rotational speed of magnetic stirrer in horizontal sample configuration. All experiments were carried out in triplicates. Figure 4.6 shows that deposit removal were ranging between 1.17% (± 0.5%) and 19.51% (±0.5%). After 120 minutes of experiment, samples soaked in water at 80°C stirred magnetic stirrer at 400 rpm gives the highest deposit removal (19.51 ± 0.05%) while samples soaked in water at 20°C without stirring gives the lowest deposit removal (1.17 ± 0.05%). It also clearly shows that the deposit removal is a function of temperature and stirring speed.

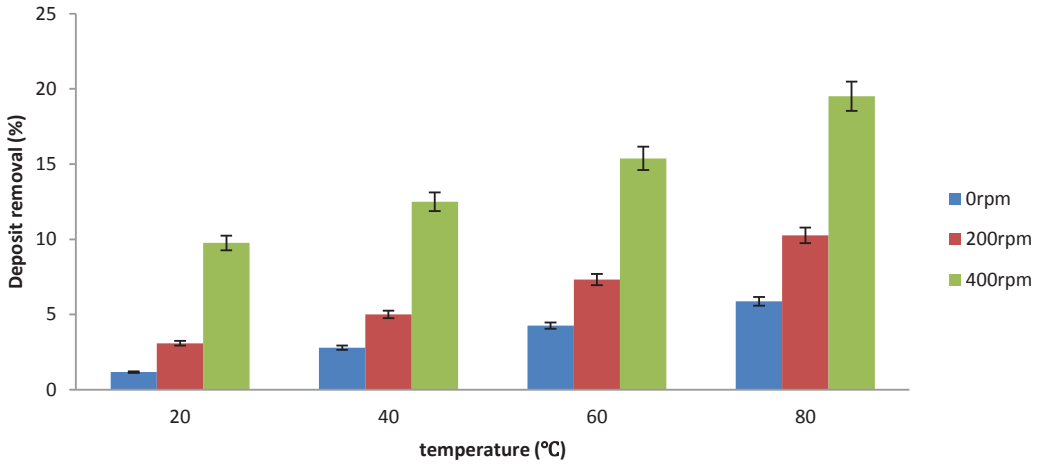


Figure 4.6: Deposit removal of egg yolk deposit soaked in water at different temperatures and stirring conditions

4.1.7 Removal of egg yolk deposits soaked in sodium hydroxide solution at different stirring conditions and temperatures

In this section, deposit removal of egg yolk deposits soaked in sodium hydroxide solution was investigated. Samples were soaked in sodium hydroxide solution at two different concentrations, four different temperatures and three different stirring speeds. All experiments were carried out in triplicates. Figure 4.7(a) shows egg yolk deposits removal for samples soaked in 0.5% w/w sodium hydroxide solution at 20, 40, 60, and 80^{o}C with 200 and 400 rpm stirring speed. After 120 minutes of experiments, deposit removal were ranging between 9.37% ($\pm$ 0.5%) and 100%. It also shows that samples soaked in 0.5% w/w sodium hydroxide solution at 80^{o}C with 200 and 400 rpm gave the highest deposit removal (100%) while samples soaked in 0.5% w/w sodium hydroxide solution at 20^{o}C without stirring gave the lowest deposit removal (9.37 $\pm$ 0.5%). 100% removal were achieved between 67 and 80 minutes for samples soaked in 1% w/w sodium hydroxide solution at 80^{o}C with 200 and 400 rpm stirring speed.

Figure 4.7(b) shows egg yolk deposits removal for samples soaked in 1% w/w sodium hydroxide solution at 20, 40, 60, and 80^{o}C with 200 and 400 rpm stirring speed. After 120 minutes of experiments, deposit removal were ranging between 10.17% ($\pm$ 0.5%) and 100%. It also shows that samples soaked in 1% w/w sodium hydroxide solution at 80^{o}C with 200 and 400 rpm gave the highest deposit removal (100%) while samples soaked in 1% w/w sodium hydroxide solution at 20^{o}C without stirring gave the lowest deposit removal (10.17 $\pm$ 0.5%). 100% removal were achieved between 59 and 70 minutes for samples soaked in 1% w/w sodium hydroxide solution at 80^{o}C with 200 and 400 rpm stirring speed. It was clearly showed from both figures that egg yolk deposit

removal was controlled by temperature and stirring condition.

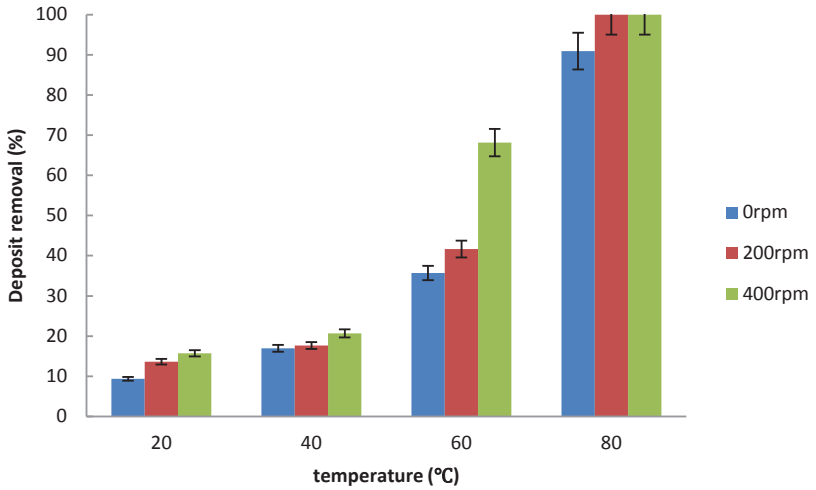


Figure 4.7(a): Removal of egg yolk deposit samples soaked in 0.5% w/w sodium hydroxide solution at different temperatures and stirring speed

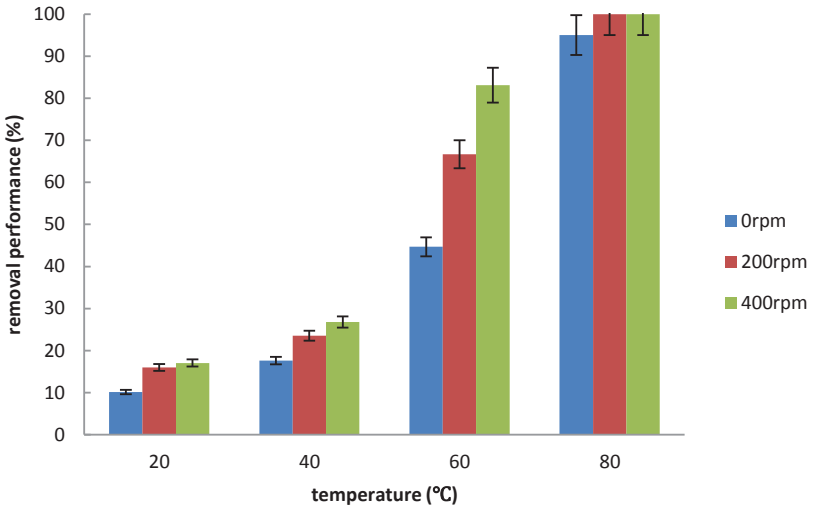


Figure 4.7(b): Removal of egg yolk deposit samples soaked in 1% w/w sodium hydroxide solution at different temperatures and stirring speeds

4.2 Discussions

4.2.1 Effect of chemical concentration on swelling performance

Pérez-Mohedano *et al*., (2016) have used FDG to determine egg yolk deposit thickness. In this study, they have found that increasing in chemical concentration and temperature resulted in increasing of swelling and water uptake. The results also indicate that chemical concentration role is important than the temperature of the cleaning solution. Some localised blisters were observed on the on the surface of the egg yolk deposit after 2 hours of experiment which were believed as the effect of hydrolysis reactions due to high alkalinity. Saikhwan et al., (2010) stated in the previous paper that the high concentration of hydroxyl ions helps to break down the peptide bonds (cohesive failure) which leading to weakened network strength. Higher in deposit mass and variability after 150 minutes of experiment shown in the gravimetric tests were explained by the presence of big blisters, which help to diffuse the solvent into the network (Pérez-Mohedano *et al*.,2016).

Swelling happened once protein-type materials contacted with alkaline substances (Saikhwan et al., 2010). One mechanism proposed by Jeurnink and Brinkman (1994) suggested that the major factor of swelling was the adsorbing of water; meanwhile alkali just helps reinforcing the process. Whereas the results of this research indicated that it was alkali that dominated swelling process. By comparing Figure 4.5 (a), (b), and (g), it was found that samples soaked in higher chemical concentration have more significant and quicker swelling event i.e. for samples soaked in water, 0.5% and 1% w/w sodium hydroxide at 40℃, without stirring, the maximum deposit swelling performance were 116%, 149.2% and 201.8% respectively, the time of which took to reach the maximum

were 90, 90 and 30 minutes (Figure 4.8(b)).

Higher concentration of alkaline solutions means greater pH values, with 12.96 for 0.5% w/w sodium hydroxide solution and 13.86 for 1% w/w sodium hydroxide solution. In this study, for each kind of protein deposit, swelling only happened in a certain range of pH values, and the highest performance was achieved at the optimum swelling pH value (Mercadé-Prieto et al., 2007). Mercadé-Prieto *et al*. (2007) has identified a pH threshold, below which swelling would not take place, and a suppressing pH, over which the swelling was suppressed. It was shown in Figure 4.5 (b), (c), (d), (e), and (f) that before reaching the optimum swelling pH, the swelling performance increased rapidly, and after the optimum pH, it decreased significantly. It was very important to identify the swelling pH range and the optimum pH for each protein deposit, because generally more swelling led to more open and more readily removable form resulting in better removal effects (Fryer et al., 2005).

This is aligned with the present study which shows that chemical concentration affect swelling of egg yolk deposits.

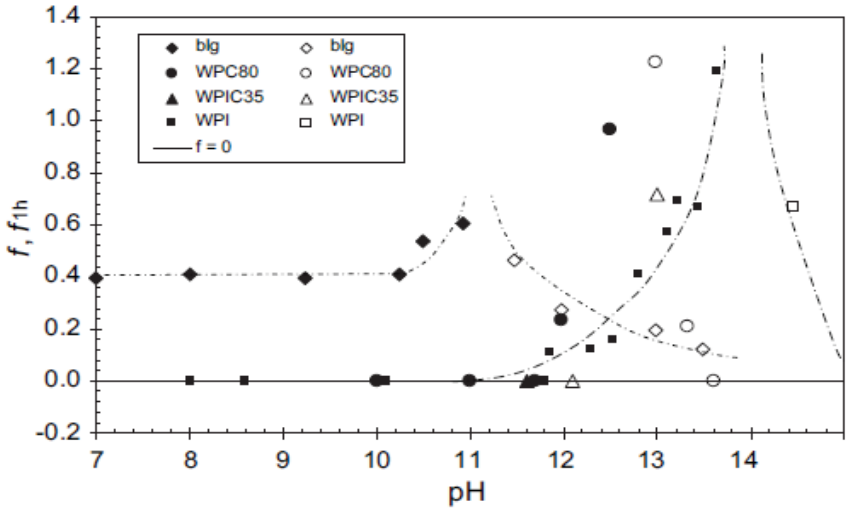


Figure 4.8(a): Effect of pH on swelling behaviour of β-lactoglobulin deposit and WPI (whey protein isolate) at 20℃. X axis represented swelling performance. Solid symbols – no dissolution; open symbols – dissolution observed (Mercadé-Prieto et al., 2007).

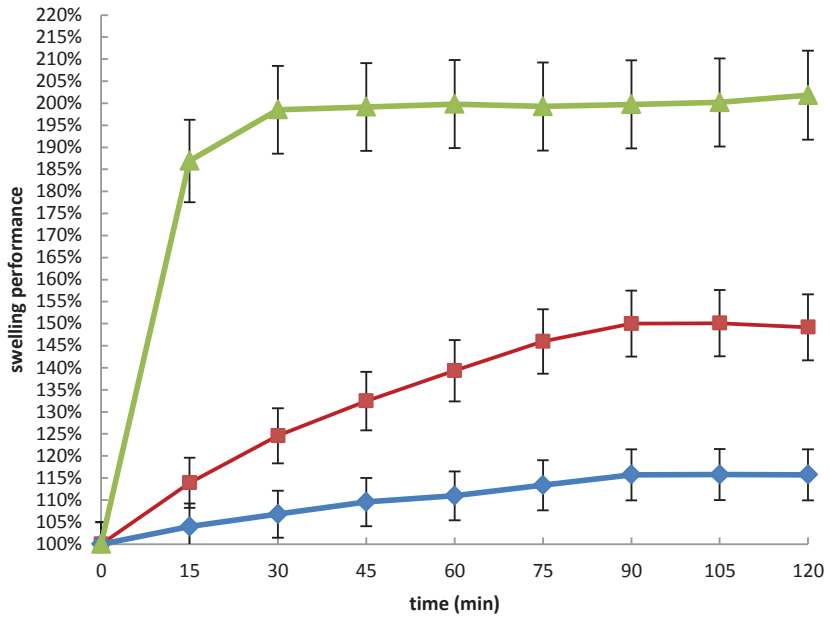


Figure 4.8(b): Swelling performance of egg yolk deposit samples soaked in water, 0.5% w/w sodium hydroxide solution, and 1% w/w sodium hydroxide solution at 40°C without stirring

4.2.2 Effect of different temperature on swelling performance of egg yolk deposit

Figure 4.5 (a) shows the swelling performance of water without stirring at three different temperatures. The trends for all experiments look quite similar, i.e. the swelling performance increase over time, with the maximum performance ranging between 115 and 116%. It also shows that the swelling of egg yolk deposit in water did not highly affected by temperature. In Figure 4.5 (b), similar trend lines were obtained for the swelling of samples soaked in 0.5% w/w sodium hydroxide solution without stirring at 40 and 60℃, in which a plateau region was reached and a maximum value was achieved

between 90 and 105 minutes. However for 80℃, the graph did not increase as fast as the other two experimental conditions, and the plateau value was significantly lower than the other two. This was explained by 90.91% deposit removal was achieved after 120 minutes at this condition, which means the removal has dominated the process before the deposit swelled sufficiently. The best swelling temperature for both alkaline solutions at both stirring conditions were 40℃, except for 0.5% sodium hydroxide without stirring where 40 and 60℃ could reach comparable maximum swelling performance (149.2% and 152% respectively). This happens because of more significant stirring and greater temperature which leads to more significant removal, thus lower swelling performance is shown.

4.2.3 Balance between swelling and removal of deposits

Since swelling and removal occur simultaneously, there is a balance between them (Fryer et al., 2005). When deposit removal predominated the event, deposit swelling would be declined. Figure 4.9 shows the swelling performance of samples soaked in 1% w/w sodium hydroxide in three different conditions. The removal performance increase (from 17.7% to 41.7%, as shown in Figure 4.6 as the temperature increase at 200 rpm from 40 (blue curve) to 60℃ (red curve), however the swelling performance declines as the temperature increase. The red curve was underneath the blue curve representing it lower rate and less sufficiently. Similarly, by comparing the red and the green curve, when stirring was introduced, more removal and less swelling were achieved, with red curve was underneath green curve which means slower and less complete swelling occurred. Thus, it explains why the samples soaked in both 0.5 and 1% w/w sodium hydroxide solutions at 80℃ with 200 rpm stirring shows 100% deposit removal within

120 minutes, and decrease in swelling performance after 15 minutes soaking which shown in Figure 4.5 (h) and (j), demonstrating that deposit removal dominated the process.

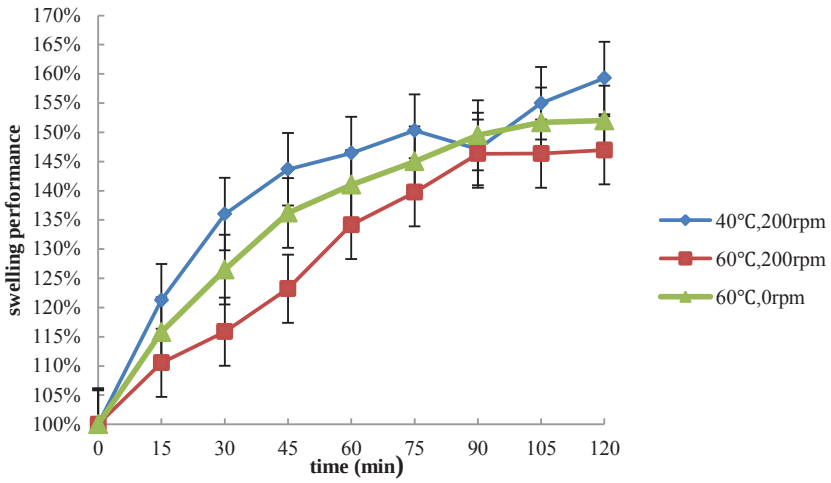


Figure 4.9: Swelling performance of egg yolk deposit soaked in 1% w/w sodium hydroxide solution at 3 different temperatures without stirring and with 200 rpm stirring

4.2.4 Effect of different chemical concentrations, temperatures and stirring speeds on deposit removal

Christian and fryer (2003) identified the factors that affect the cleaning of proteinaceous dairy deposits are temperature, fluid flow, chemical concentration, chemical type and the amount of deposits present. In the cleaning of a type 3 deposit, shear force of water alone is not enough to remove the deposit, thus expensive chemicals are required to remove the deposits. The complex inter-relationship between all factors makes it difficult for us to determine the exact role of each factor. However, the cleaning fluid motion is needed to transport the cleaning agent to the deposit surface interface, to remove the

deposit from the surface and to transport the removed deposit out of the system while the temperature is also crucial as it helps to increase the rate of reaction and the diffusion of cleaning chemicals when the temperature is increase and as well as modify the viscosity of the cleaning chemical fluid. It also found that cleaning chemical plays an important role in the swelling of the deposit (Christian and Fryer, 2003).

Temperature is the most important factor for type 3 deposit removal (Christian and Fryer, 2006). Generally, the cleaning rate will increase as the temperature increases (Hankinson and Carver, 1968; De Goederen et al., 1989; Fryer and Bird, 1994; Alfa Laval, 2003). Gillham (1999) reported that cleaning of whey protein foulants from stainless steel surface is highly controlled by temperature. The swelling phase is not really affected by temperature even though the rest of the cleaning process depends on temperature. The decay phase becomes shorter significantly as the deposit-liquid temperature increase beyond 50^{o}C. Aziz (2008) stated that increase in temperature results in decrease in cleaning time. No deposit removal was observed for whey protein concentration (WPC) deposits at 30^{o}C at any flow rate or sodium hydroxide concentration tested. This can be concluded that WPC deposit requires a high temperature (>50^{o}C) to be removed. This agrees with the findings from this research which shows that increasing temperature leads to increase in deposit removal. The egg yolk deposits soaked in water without stirring show increase in deposit removal when the temperature is increased from 20 to 80^{o}C (figure 3.3.1). Complete deposit removal is given by the samples soaked in sodium hydroxide solution at 80^{o}C with stirring at 200 and 400 rpm (figure 3.3.2 (a) and (b)).

Stirring is introduced in this study to provide shear stress (mechanical force) to the

cleaning system. Fryer and Asteriadou (2009) stated that more shear stress would result in better cleaning effects. Othman et al (2010) conducted studies to investigate the cleaning of sweet condensed milk (SCM) using a flow cell found that increasing the flow rate from 0.25 to 0.5 ms^{-1} reduced the cleaning time of SCM. Gordon et al (2012) reported that doubling the shear stress applied using scanning fluid dynamic gauge reduced the cleaning time of egg yolk deposit layer by approximately 20%. Perez-Mohedano (2014) found that applying shear stress using scanning fluid dynamic gauge reduced the cleaning time of egg yolk deposit immersed in buffered protease. According to the data obtained from this study, stirring (shear stress) helps to increase deposit removal of egg yolk deposit soaked in water (without chemical action) with samples soaked in water at 400 rpm stirring speed give the highest deposit removal (figure 3.31). These findings seem to be in agreement with the findings from the previous researchers which suggested that increase in shear stress leads to increase in deposit removal.

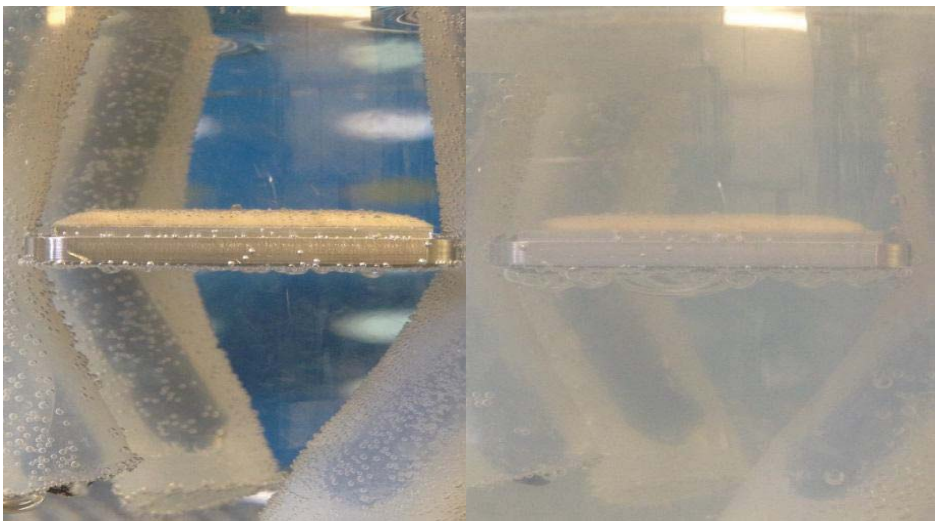
As a type 3 deposit, egg yolk requires chemical action to be cleaned. Othman et al (2010) stated that increasing sodium hydroxide solution concentration from 0.5 to 1.5% does not really affect the cleaning time of sweet condensed milk (SCM) deposit. Therefore, they came up with a conclusion that the optimum concentration of sodium hydroxide solution is 0.5%. Gordon et al (2012) conducted studies on cleaning of egg yolk deposit using scanning fluid dynamic gauge reported that no deposit removal is shown after 400 minutes of experiment without protease. When protease is introduced into the system, deposit swelling is interrupted after 40 minutes of experiments and deposit starts to remove after 60 minutes of experiment. Deposit is totally removed after 140 minutes of experiment. Perez-Mohedano et al (2014) also observed no egg yolk

deposit removal after 150 minutes soaking in buffer solution. Deposit swelling starts to decline after 30 minutes of experiment when protease is introduced into the cleaning system. Complete deposit removal is observed after 100 minutes of experiment with protease. Perez-Mohedano et al (2016) stated that increase in temperature and pH of hydration fluid led to increase in swelling of egg yolk deposit. However, results suggest that no significant effect of temperature is observed for experiment at low pH which means that the effect of pH (chemical action) is greater than temperature. In the present studies, egg yolk deposits soaked in sodium hydroxide solution at 0.5 and 1% concentration have higher deposit removal (figure 3.3.2 (a) and (b)) than those deposits soaked in water at the similar experimental conditions (figure 3.3.1) with complete deposit removal shown by the samples soaked in sodium hydroxide solution at 80^{o}C stirred using magnetic stirrer at 200 and 400 rpm. This agrees with the findings from previous workers which showed that chemical action plays an important role in cleaning of deposit.

4.2.5 Removal of egg yolk deposits

In most cases, cohesive removal and adhesive removal happened simultaneously, which could not be differentiated (Saikhwan et al., 2010). The removal of egg yolk deposit was from the external layer to the internal layer gradually. The swelling and removal stage were indistinguishable. Figure 4.10 shows that the hydrating solution became cloudy after 45 minutes of experiment with 5% w/w sodium hydroxide solution at 200 rpm stirring speed and 60℃ temperature, indicating that the removal has happened. While according to Figure 4.5 (i), after 45 minutes of the experiment, the swelling diagram has not reached the plateau region yet, which means it would continue

to swell afterwards, thus, indicates that deposit swelling and removal occurs at the same time.



(a) (b)

Figure 4.10: Pictures of egg yolk deposit samples soaked in 1% w/w sodium hydroxide solution at 60℃ with 200 rpm stirring at (a) 0 minute (b) 45 minutes

4.2.6 Cleaning mechanism of egg yolk deposits

In this study, it is found that temperature is the most important for egg yolk deposit removal. The whole procedure can be divided into different phases. At the early stage of cleaning, the detergent diffused from the bulk fluid to the deposit and diffused inside the deposit (Saikhwan et al., 2010). Diffusion requires energy and was highly dependent on temperature. In the meantime, reaction occurs between deposit and detergent, which breaks the firm structures of the protein gel, which leads to swelling (Saikhwan et al., 2010). Generally, increase in deposit swelling results in more open form of deposit and more removals. The pH value is found to be the dominant factor for swelling (Mercadé-

Prieto et al., 2007). After the swelling form is formed, the deposit becomes more sheared off rather than dissolved which leads to deposit removal (Christian and Fryer, 2006). It can be concluded that the swelling of deposit is controlled by chemical action and temperature while the removal of deposit is controlled by temperature and mechanical action (shear stress).

Saikhwan et al. (2010) also stated the mechanism that might explain the effect of temperature on deposit removal. Protein structure could be regarded as polyelectrolyte polymers where large or small aggregates interacted with each other in a net structure by covalent (intermolecular disulfide bonds) and non-covalent (i.e. hydrophobic and intermolecular β-sheets) bonds. This type of structure requires high temperature to break which allow the swelling and removal to occur. This agrees with the data collected which indicates that temperature increase the swelling and the removal of egg yolk deposit.

4.2.7 Comparison on egg yolk swelling performance between previous and present studies

It is well understood that swelling is part of the cleaning process. *Liu et al (2007)* stated that swelling occurs when whey protein concentration (WPC) deposit soaked in NaOH at different concentration. *Christian* and *Fryer* (2003) also reported that swelling of WPC deposit during intermediate section of WPC cleaning using plate heat exchanger (PHE) was found to be the effect of chemical action. *Perez-Mohedano et al* (2016) stated that increase in temperature and pH of hydration fluid led to increase in swelling of egg yolk deposit. However, it was found that the effect pH (chemical action) was greater than

temperature. *Gordon et al*. (2012) found that chemical action (protease) and mechanical action (shear stress) suppress the swelling of egg yolk deposit layer soaked in buffer solution and reduced the final deposit thickness through deposit removal. A research by Saikhwan et al (2010) also stated that swelling was controlled by pH of the hydration fluid. This agrees with the present results on hydration of egg yolk deposit which shows that pH (NaOH concentration) controls the swelling performance of egg yolk deposit (figure 4.5 (a) – (i)). Results from Gordon et al (2012) Perez-Mohedano (2014); Perez-Mohedano (2016) on swelling performance of egg yolk deposit using scanning fluid dynamic gauge show that in the presence of chemical action, swelling increase rapidly during the first 30 minutes of experiment which seen to be agreed with the recent studies that show swelling of egg yolk deposit increase rapidly during the first 20 minutes of experiment at room temperature (figure 4.1 – 4.4) while at 40 and 60 ^{o}C, swelling was found to be increased rapidly during the first 30 minutes of experiment (figure 4.1).

4.2.8 Novelty and innovations

The hydration experiment set up was exclusively designed to study the effect of temperature on the swelling and the cleaning of egg yolk deposit during hydration. Magnetic stirrer and mixing impeller is used in this set up to provide mechanical force while hot plate and thermometer are used to control the temperature throughout the experiment. Results from the experiment were obtained by measuring the thickness, moisture content and the percentage of deposit removal. Egg yolk powder is used to prepare the deposit samples in order to study the swelling and the cleaning of a fat

based deposit. According to the results obtained in this research, temperature plays a major role in the swelling and the cleaning of egg yolk deposit. Deposits were seen to be totally removed at 80^{o}C while no complete removal of deposits was shown at 40^{o}C and 60^{o}C. Swelling performance of egg yolk deposits soaked at 80oC hydration was found to be decreased because swelling and deposit removal happens simultaneously during the experiment.

4.3 Conclusions

These hydration and cleaning experiments can be used to study the effect of different hydrating solutions, temperatures, sample configurations and mixing/stirring conditions on the hydration of egg yolk fouling deposit on stainless steel surface in terms of the moisture content of the deposit, swelling performance, and removal and cleaning of the egg yolk deposit fouled on the stainless steel surface.

Basically, hydration of egg yolk deposit was affected by different hydrating solutions (water, sodium hydroxide solution, LAS solution and detergent solution), different hydration pH, different hydration temperature (20, 40, 60 and 80^{o}C), different sample configurations (vertical and horizontal) and different stirring conditions. After 120 minutes of hydration, samples soaked in 1% w/w sodium hydroxide solution have the highest moisture content in all cases followed by samples soaked in 1% w/w LAS solution, 1% w/w detergent solution and samples soaked in water. Results from the experiments also indicate that, in all cases there is a slightly significant difference in moisture content between samples soaked horizontally and samples soaked vertically in hydrating solution. Higher stirring speed also gives higher moisture content in all cases.

The deposit thickness and swelling can be measured by the image processing technique. Results also indicate that deposit thickness and swelling were affected by different chemical concentrations, hydrating temperatures, and stirring conditions. In some cases, higher chemical concentration gives higher swelling performance, higher temperature shows higher swelling performance and higher stirring speed gives slightly higher swelling performance.

The removal and cleaning of the egg yolk fouling deposit can be determined by the deposit removal after 120 minutes of experiment. Results from the experiments suggest that the egg yolk deposit removal was affected by different hydrating temperatures, chemical concentrations and stirring conditions. In all cases, higher chemical concentration gives higher swelling performance, higher temperature shows higher swelling performance and higher stirring speed gives slightly higher swelling performance. All samples soaked in sodium hydroxide solution at 80^{o}C with 200 rpm and 400 rpm stirring showed 100% deposit removal, which means deposit, were totally cleaned off.

Generally, these methods and techniques can be used to study the hydration and cleaning of the egg yolk fouling deposit and more works will be carried out in future using cleaning rig and micromanipulation rig in order to study more about the cleaning behaviour.

Removal of swollen deposit by dissolution and shear is also important in determining the overall cleaning time.

CHAPTER 5: THE EFFECT OF CHEMICAL AND MECHANICAL ACTION ON SWELLING AND REMOVAL OF EGG YOLK DEPOSITS DURING HYDRATION AT ROOM TEMPERATURE

5.1 Results

5.1.1 Moisture content of egg yolk deposits soaked in different cleaning solutions

In this section, experiments on hydration of egg yolk deposit in different cleaning solutions, at different mixing level, and sample configurations were carried out. All experiments were carried at room temperature in triplicates. Figure 5.1 (a) shows the experiments on hydration of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at horizontal sample configuration without mixing (stirring). Results clearly indicate that samples soaked in 1% w/v sodium hydroxide show the highest moisture content (59.3 $\pm$ 1 %) after 120 minutes of experiment, followed by samples soaked in 1% LAS solution (52.5 $\pm$ 1 %), 1% detergent solution (46.53 $\pm$ 1 %) and water (33.33 $\pm$ 1 %). It also found that all samples soaked in all cleaning solutions are going through the similar trend as the moisture content is found to be rapidly increased during the first 20 minutes of experiment. Results also indicate that all trends reach plateau after 100 minutes of experiment.

Figure 5.1 (b) illustrates the experiments on hydration of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at horizontal sample configuration at 400 rpm stirring speed using magnetic stirrer. From the data obtained, it was found that Results that samples soaked in 1% w/v sodium hydroxide show the highest moisture content (61.9 $\pm$ 1 %) after 120 minutes of

experiment, followed by samples soaked in 1% LAS solution (54.39 ± 1 %), 1% detergent solution (49.55 ± 1 %) and water (38.57 ± 1 %). All samples are going through the similar trend in all cases with rapid increase in moisture content happens during the first 20 minutes of experiment. All samples reach plateau after 100 minutes of hydration.

Figure 5.1 (c) shows the experiments on hydration of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at horizontal sample configuration at 400 rpm stirring speed using mixing impeller. after 120 minutes of experiment , the highest moisture content is shown by the samples soaked in 1% w/v sodium hydroxide (64.52 ± 1 %), followed by samples soaked in 1% LAS solution (60.22 ± 1 %), 1% detergent solution (52.23 ± 1 %) and water (42.19 ± 1 %). In all cases, rapid increase in moisture content occurs during the first 20 minutes of experiment and that similar trend reaches plateau after 100 minutes of hydration.

Figure 5.1 (d) illustrates the experiments on hydration of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at vertical sample configuration without mixing (stirring). The highest moisture content is shown by the samples soaked in 1% w/v sodium hydroxide (62.11 ± 1 %) after 120 minutes of hydration. This followed by samples soaked in 1% LAS solution (50 ± 1 %), 1% detergent solution (47.14 ± 1 %) and water (35.89 ± 1 %). Similar trend in moisture content of deposit is observed in all cases with rapid increase in moisture content happens during the first 20 minutes of experiment and reach plateau throughout the last 20 minutes of the experiment.

Figure 5.1 (e) shows the experiments on hydration of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at vertical sample configuration at 400 rpm stirring speed using magnetic stirrer. It was found that samples soaked in 1% w/v sodium hydroxide give the highest moisture content (66.96 $\pm$ 1 %) after 120 minutes of experiment, followed by samples soaked in 1% LAS solution (60.27 %), 1% detergent solution (56.32 $\pm$ 1 %) and water (41.54 $\pm$ 1 %). All samples are going through the similar trend with rapid increase in moisture content occurs during the first 20 minutes of experiment and reach plateau throughout the last 20 minutes of the hydration.

Figure 5.1 (f) illustrates the experiments on hydration of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at horizontal sample configuration vertical sample configuration at 400 rpm stirring speed using mixing impeller. Results from the data obtained show that samples soaked in 1% w/v sodium hydroxide have the highest moisture content (73.61 $\pm$ 1 %) after 120 minutes of experiment. This followed by samples soaked in 1% LAS solution (64.65 $\pm$ 1 %), 1% detergent solution (58.89 $\pm$ 1 %) and water (46.43 $\pm$ 1 %). Similar trend in the moisture content of deposit is observed in all cases with rapid increase in moisture content happens during the first 20 minutes of hydration and reach plateau after 100 minutes of hydration.

These results indicate that the introduction of different cleaning chemical helps to increase the moisture content of egg yolk deposit with sodium hydroxide 1% w/v gives the highest impact on the moisture content of egg yolk deposit.

.5.1.2 Moisture content of egg yolk deposits soaked at different stirring conditions

Sets of experiment were carried out in order to investigate the effect of different stirring conditions on hydration of egg yolk deposit. For those experiments using water as the cleaning agent, samples soaked in water stirred using mixing impeller give the highest moisture content (42.19 $\pm$ 1 %) after 120 minutes of experiment, followed by samples soaked in water stirred using magnetic stirrer (38.57 $\pm$ 1 %), and samples soaked without stirring (33.33 $\pm$ 1 %). All samples are going through the similar trend with moisture content rapidly increase during the first 20 minutes of experiments.

Results from the experiments indicate that experiments using sodium hydroxide solution, LAS solution and detergent solution are also going through the similar behaviour. Samples soaked in sodium hydroxide solution, LAS solution and detergent solution stirred using mixing impeller have the highest moisture content (64.52 $\pm$ 1, 60.22 $\pm$ 1 and 52.23 $\pm$ 1 %) compare to those samples soaked in sodium hydroxide solution, LAS solution and detergent solution stirred using magnetic stirrer (61.9, 51.39 and 49.55 %) and samples soaked in sodium hydroxide solution, LAS solution and detergent solution without mixing (59.3 $\pm$ 1, 52.5 $\pm$ 1, and 47.5 $\pm$ 1 %).

Without chemical action, stirring helps to increase the percentage of moisture content by 3 to 29.37 % with samples soaked in water stirred by mixing impeller give the highest moisture content increase.

5.1.3 Moisture content of egg yolk deposits soaked in cleaning solutions at different sample configurations

Apart from the effect of different cleaning agents and mixing levels, some experiments were also carried out in order to study the effect of different sample configurations i.e horizontal and vertical configuration.

Results from this set of experiments show that after 120 minutes of experiment, all samples soaked in all cleaning agents tested (water, sodium hydroxide solution, LAS solution and detergent solution) at vertical configurations give higher moisture content than (35.89 $\pm$ 1, 62.11 $\pm$ 1, 50 $\pm$ 1, and 47.14 $\pm$ 1 %) those samples soaked at horizontal configuration (33.33 $\pm$ 1, 59.3 $\pm$ 1, 52.5 $\pm$ 1, and 47.5 $\pm$ 1%). All samples are going through the similar trend with rapid moisture content increase during the first 20 minutes of experiments.

There is only a slight difference in moisture content between samples soaked at horizontal configuration and samples soaked at vertical configuration (up to 8%) with no significant difference for samples soaked in detergent solution at vertical and horizontal configuration (47.14 $\pm$ 1 and 47.5 $\pm$ 1 %).

Results from this set of experiment also indicate that different sample configuration does not affect much on the moisture content egg yolk deposit and higher moisture content is shown in those experiments with samples soaked in water, sodium hydroxide solution and LAS solution at vertical configuration.

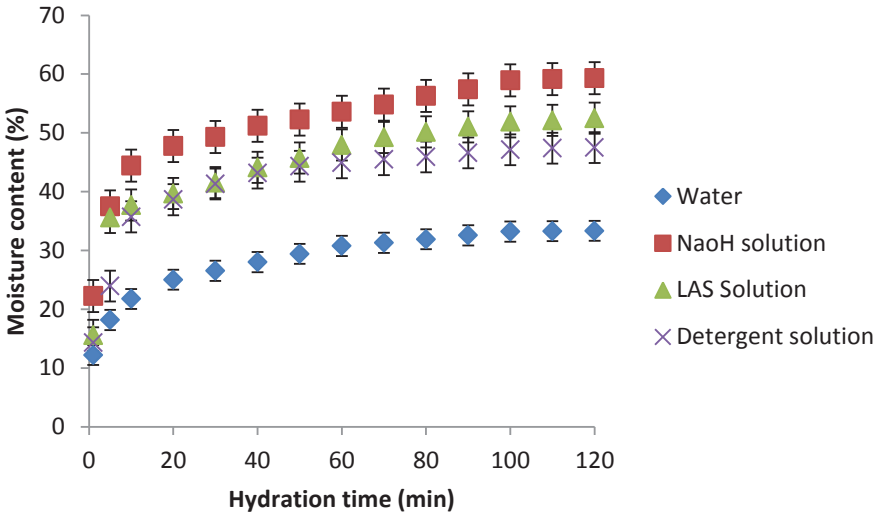


Figure 5.1 (a): Moisture of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at horizontal sample configuration without stirring

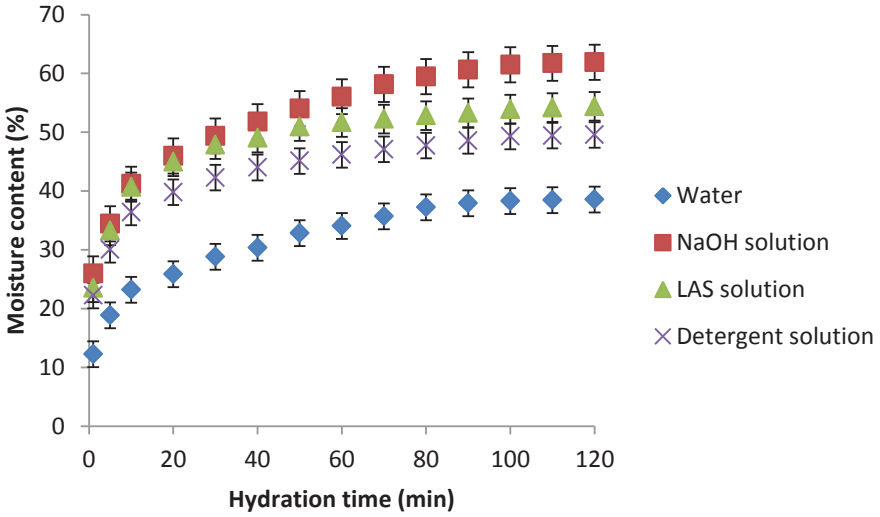


Figure 5.1 (b): Moisture content of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at horizontal sample configuration at 400 rpm stirring using magnetic stirrer

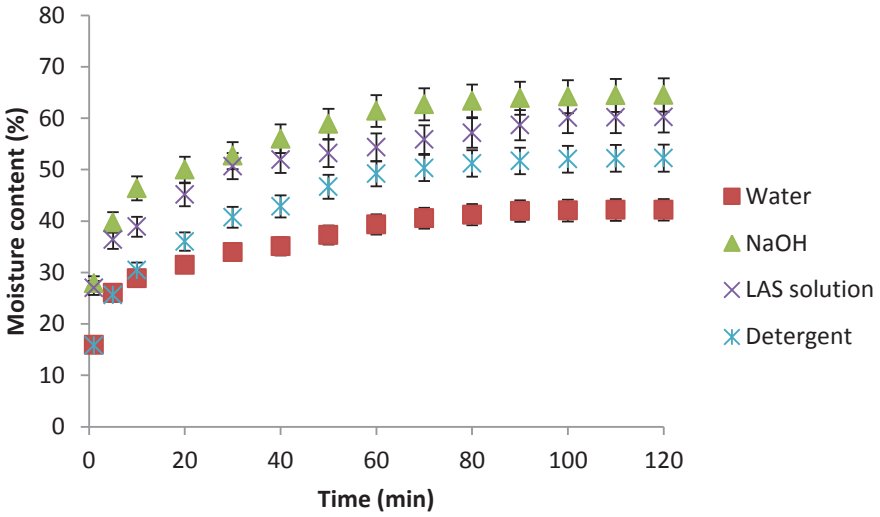


Figure 5.1 (c): Moisture content of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at horizontal sample configuration at 400 rpm stirring using mixing impeller

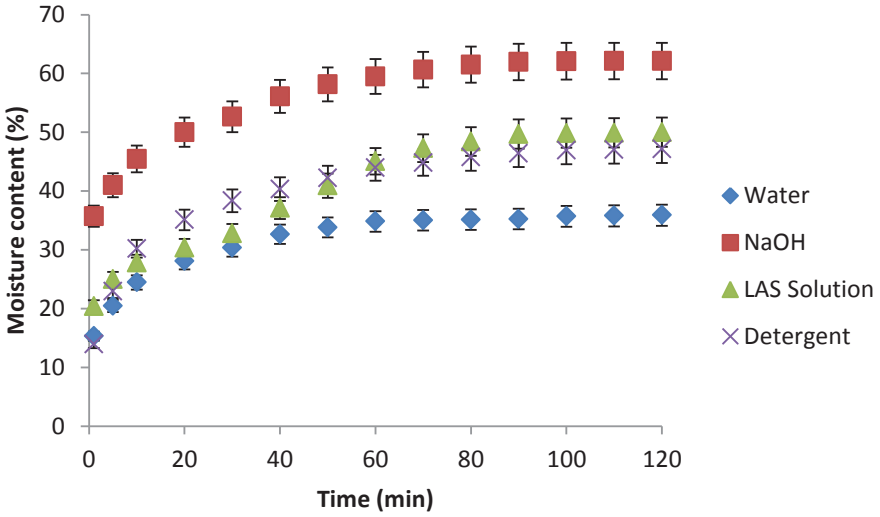


Figure 5.1 (d): Moisture content of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at vertical sample configuration without stirring

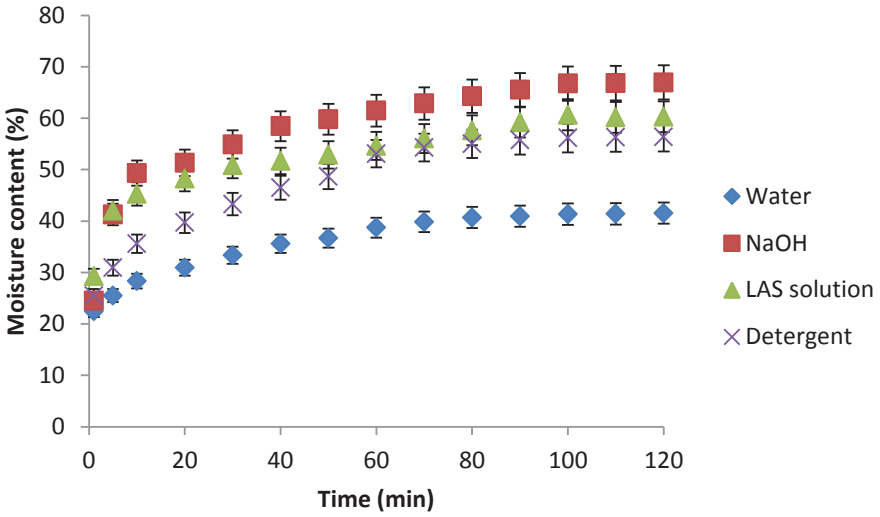


Figure 5.1 (e): Moisture content of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at vertical sample configuration at 400 rpm stirring using magnetic stirrer

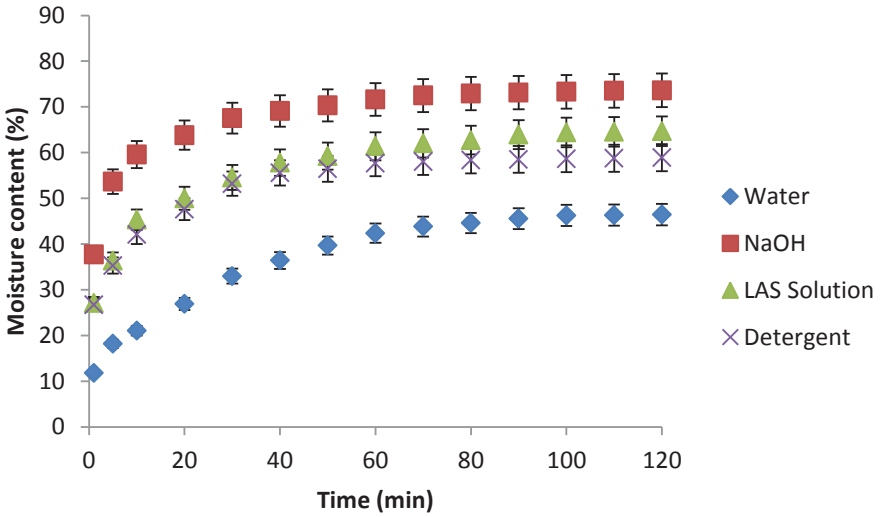


Figure 5.1 (f): Moisture content of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at vertical sample configuration at 400 rpm stirring using mixing impeller

5.1.4 Swelling performance of egg yolk deposit soaked in different cleaning solutions

In this section, experiments to study the swelling performance of egg yolk deposits were carried out in triplicates. Figure 5.2 (a) shows the experiments on swelling performance of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at horizontal sample configuration without mixing (stirring). The highest swelling performance is shown by the samples soaked in 1% w/v sodium hydroxide (30.77 $\pm$ 1 %) after 120 minutes of experiment, followed by samples soaked in 1% LAS solution (27.54 $\pm$ 1 %), 1% detergent solution (22.39 $\pm$ 1 %) and water (16.39 $\pm$ 1 %). It also found that all samples soaked in all cleaning solutions are going through the similar trend in swelling performance. Swelling performance is shown to be rapidly increased during the first 20 minutes of experiment and reaches plateau after 100 minutes of experiment.

Figure 5.2 (b) illustrates the experiments on swelling performance of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at horizontal sample configuration at 400 rpm stirring speed using magnetic stirrer. It is shown by the data obtained that the samples soaked in 1% w/v sodium hydroxide give the highest swelling performance (36.59 $\pm$ 1 %) after 120 minutes of experiment, followed by samples soaked in 1% LAS solution (31.51 $\pm$ 1 %), 1% detergent solution (25 $\pm$ 1 %) and water (19.7 $\pm$ 1 %). Similar trend in swelling performance is observed with rapid increase in swelling performance occurs during the first 20 minutes of experiment and reaches plateau throughout the last 20 minutes of hydration.

Figure 5.2 (c) shows the experiments on swelling performance of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at horizontal sample configuration at 400 rpm stirring speed using mixing impeller. The samples soaked in 1% w/v sodium hydroxide are found to have the highest swelling performance (40 $\pm$ 1 %) after 120 minutes of experiment, followed by samples soaked in 1% LAS solution (33.33 $\pm$ 1 %), 1% detergent solution (31.51 $\pm$ 1 %) and water (22.06 $\pm$ 1 %). In all cases, swelling performance of egg yolk deposit is shown to have a similar trend with swelling performance is found to be increased rapidly throughout the first 20 minutes of the experiment. This similar trend reaches plateau after 120 minutes of experiment.

Figure 5.2 (d) illustrates the experiments on swelling performance of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at vertical sample configuration without mixing (stirring). Those samples soaked in 1% w/v sodium hydroxide are observed to give the highest swelling performance (32.91 $\pm$ 1 %) after 120 minutes of experiment, followed by samples soaked in 1% LAS solution (30.56 $\pm$ 1 %), 1% detergent solution (26.09 $\pm$ 1 %) and water (19.35 $\pm$ 1 %). It also found that all samples are going through a similar trend in swelling performance all cases with rapid increase in swelling performance happens during the first 20 minutes of experiment and reaches equilibrium after 100 minutes of hydration.

Figure 5.2 (e) shows the experiments on swelling performance of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at vertical sample configuration at 400 rpm stirring speed using magnetic stirrer. It is clear that the samples soaked in 1% w/v sodium hydroxide have the highest

swelling performance (40.7 ± 1 %) after 120 minutes of experiment, followed by samples soaked in 1% LAS solution (35.06 ± 1 %), 1% detergent solution (28.17 ± 1 %) and water (23.19 %). A similar trend in swelling performance of egg yolk deposit is observed in all cases with swelling performance is found to increase rapidly during the first 20 minutes of experiment, and reach plateau after 100 minutes of experiment.

Figure 5.2 (f) illustrates the experiments on swelling performance of egg yolk deposits soaked in water, 1% w/v sodium hydroxide, 1% w/v LAS solution and 1% detergent solution at horizontal sample configuration vertical sample configuration at 400 rpm stirring speed using mixing impeller. The highest swelling performance is shown by the samples soaked in 1% w/v sodium hydroxide (42.05 ± 1 %) after 120 minutes of experiment, followed by samples soaked in 1% LAS solution (36.71 ± 1 %), 1% detergent solution (33.33 ± 1 %) and water (26.76 ± 1 %). All samples are going through the similar trend with swelling performance increase rapidly during the first 20 minutes of experiment and reach equilibrium after 20 minutes of hydration.

These results suggest that the swelling performance of egg yolk deposit is controlled by the cleaning fluid with sodium hydroxide 1% w/v found to have the highest impact on the moisture content of egg yolk deposits.

5.1.5 Swelling performance of egg yolk deposit soaked at different stirring conditions

In this set of experiment, egg yolk deposits were soaked in cleaning solutions at three stirring conditions (without mixing, magnetic stirrer and mixing impeller). Magnetic stirrer was used in this experiment to provide low mixing while mixing impeller was used to provide high mixing to the cleaning solutions. It is shown in the results (figure 3.2(a) –

3.2(c)) that samples soaked in all cleaning solutions (water, sodium hydroxide solution, LAS solution and detergent solution) stirred using mixing impeller have the highest swelling performance (22.06 ± 1, 40 ± 1, 33.33 ± 1 and 31.51± 1 %) after 120 minutes of experiment, followed by samples soaked in solutions stirred using magnetic stirrer (19.70 ± 1, 36.59 ± 1, 31.51 ± 1, and 25 ± 1 %) and samples soaked in cleaning agents without stirring (16.39 ± 1, 30.77 ± 1, 27.54 ± 1, and 22.39 ± 1%).

All samples in this set of experiment are going through the similar behaviour with rapid swelling performance increase during the first 20 minutes of experiment. Results from the experiments suggest that stirring helps to increase egg yolk deposit swelling performance by up to 40% with the highest swelling performance increase are shown between the samples soaked in detergent solution stirred by mixing impeller (31.51%) and the samples soaked in detergent solution without stirring (22.39%) which give a 40.73% increase in swelling performance. Without chemical action, stirring increase the swelling performance by up to 38.29 %. Thus, it can be concluded that mechanical action of stirring affect the swelling performance of egg yolk deposits.

5.1.6 Moisture content of egg yolk deposit soaked in cleaning solutions at different sample configurations

Set of experiments were carried out to investigate to the effect of different sample configurations i.e. horizontal and vertical configurations. Samples were soaked in water, sodium hydroxide solution, LAS solution, and detergent solution at horizontal and vertical configuration for 120 minutes. Results from this set of experiment (figure 3.29(a) and figure 3.2(d)) show that there is a slight difference in swelling performance between

samples at horizontal and samples soaked at vertical configuration with samples soaked at vertical configuration (19.35 ± 1, 32.91 ± 1, 30.56 ± 1, and 32.91 ± 1 %) have higher swelling performance than samples soaked at horizontal configuration (16.39 ± 1, 30.77 ± 1, 27.53 ± 1, and 30.77 ± 1%).

Without chemical and mechanical action, the difference in swelling performance between samples soaked at both configurations is 18.06 % while with chemical action but without mechanical action, the difference in swelling performance between samples soaked at both configurations is from 6.95 to 11.01 %. All samples are going thru the similar trend with rapid increase in swelling performance occurs during the first 20 minute of experiment and reach plateau after 100 minutes of experiment.

Results also indicate that different samples configuration affect the swelling performance of egg yolk deposit with vertical sample configuration gives higher swelling performance than horizontal samples configuration.

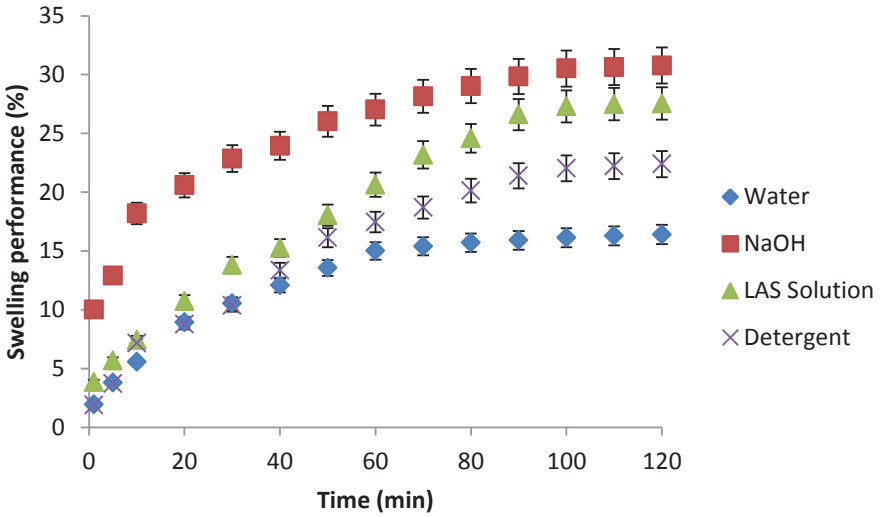


Figure 5.2(a): Swelling performance of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at horizontal sample configuration without stirring

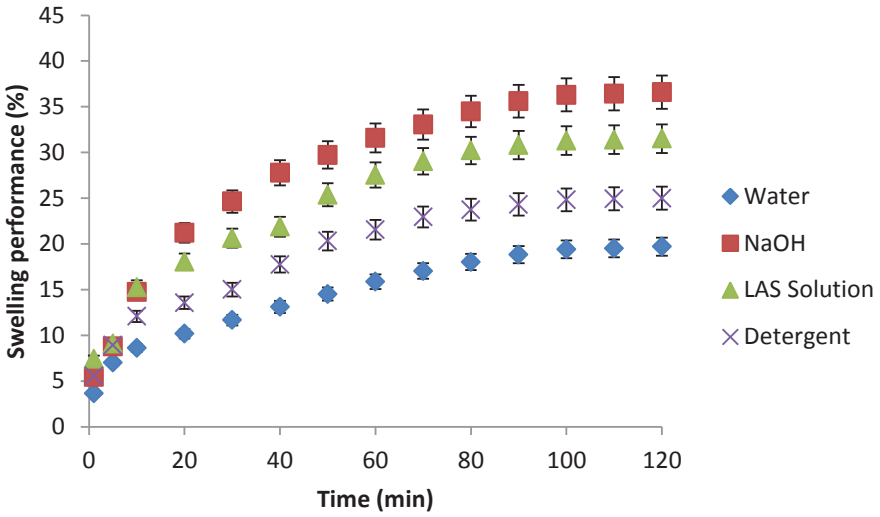


Figure 5.2 (b): Swelling performance of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at horizontal sample configuration at 400 rpm stirring using magnetic stirrer

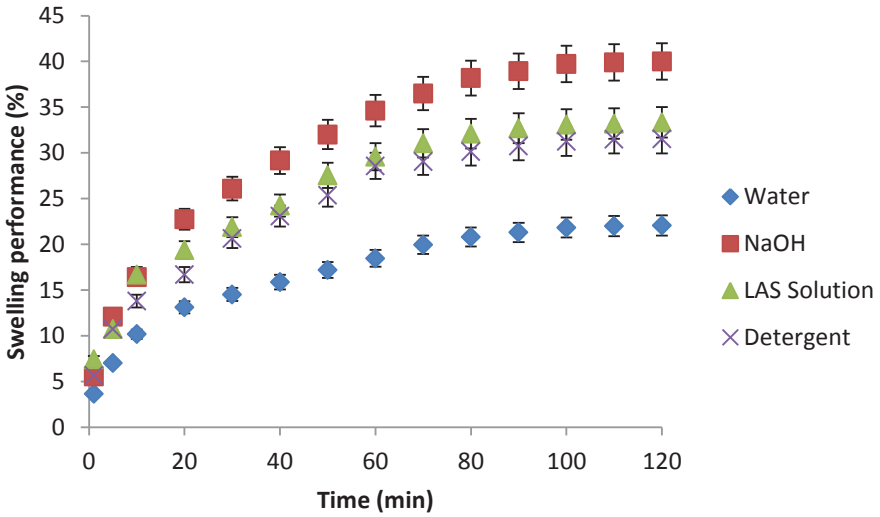


Figure 5.2 (c): Swelling performance of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at horizontal sample configuration at 400 rpm stirring using mixing impeller

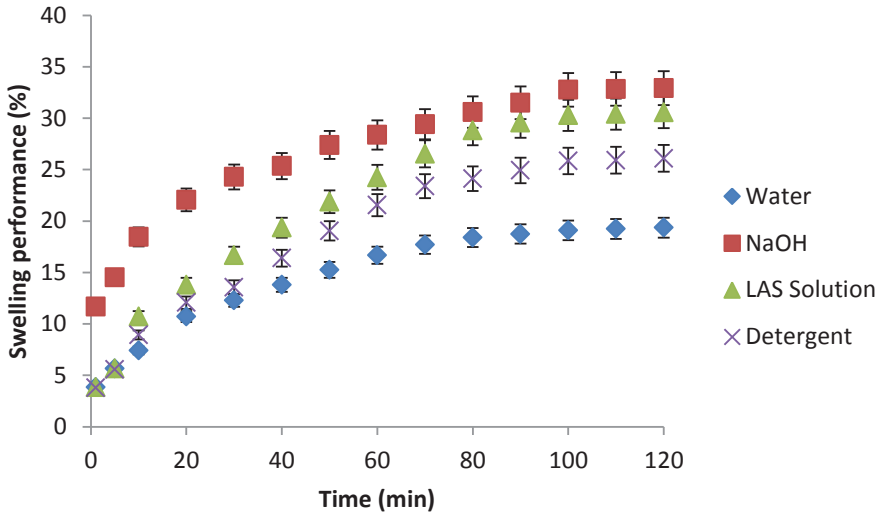


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Figure 5.2 (d): Swelling performance of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at vertical sample configuration without stirring

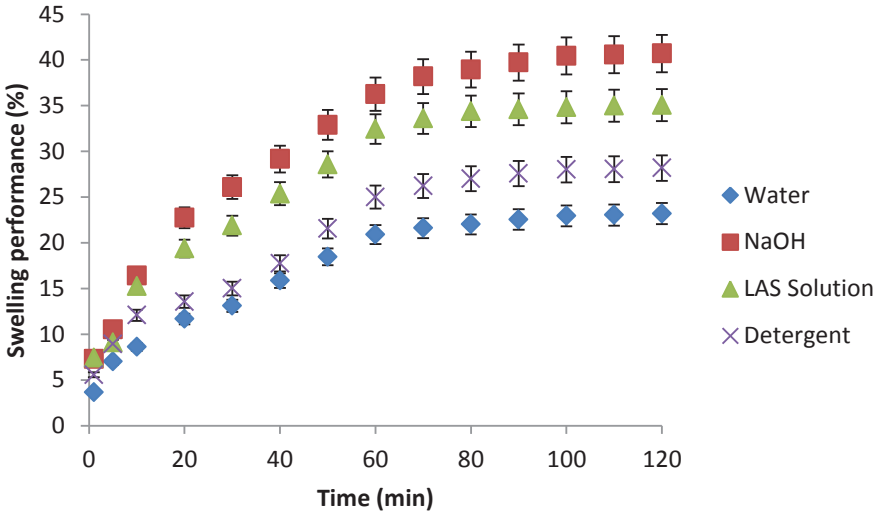


Figure 5.2 (e): Swelling performance of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at vertical sample configuration at 400 rpm stirring with magnetic stirrer

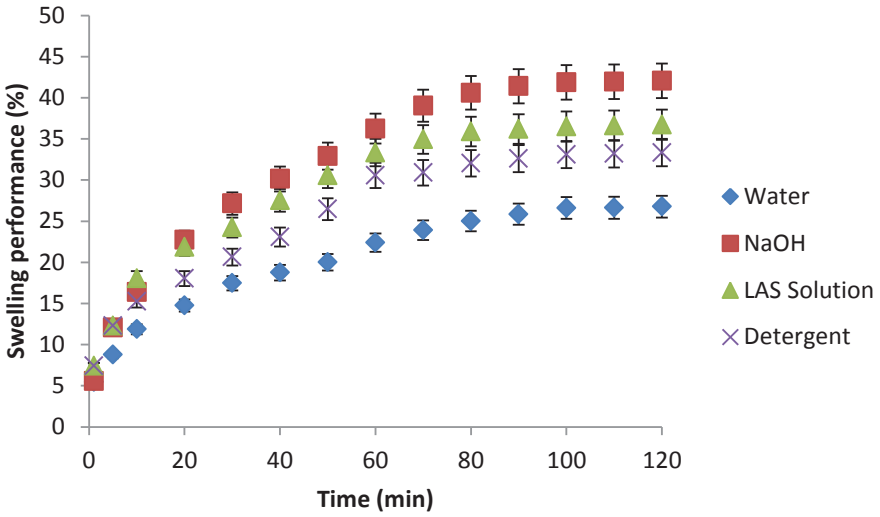


Figure 5.2 (f): Swelling performance of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution in vertical sample configuration at 400 rpm stirring with mixing impeller

5.1.7 Removal of egg yolk deposit soaked in different cleaning solutions

Series of experiment was carried out to investigate the effect of different cleaning solutions on the deposit removal of egg yolk deposit. In this set of experiment, egg yolk deposits were soaked in water, sodium hydroxide solution LAS solution, and detergent solution for 120 minutes. Results (figure 5.3(a) and 5.3(b)) indicate that all samples soaked in sodium hydroxide solutions give the highest deposit removal (10.81 $\pm$ 1 %), followed by those samples soaked in LAS solution (9.81 $\pm$ 1 %), samples soaked in detergent solution (7.31 $\pm$ 1 %), and samples soaked in water (6.98 $\pm$ 1 %).

Different cleaning solutions were used to provide chemical actions for the cleaning of egg yolk deposit, since egg yolk is classified as type 3 deposit by *Fryer* and *Asteriadou* (2009) which required chemical action to clean. Sodium hydroxide solution was found to be the best chemical used to clean the deposit with 55.01 % increase in deposit

removal compares to deposit soaked in water, while samples soaked in LAS solution have 40.75 % deposit removal increase and samples soaked in detergent solution helps to raise the deposit removal by 5.02 %. Results also prove that different cleaning solutions help to provide chemical action in order to increase the cleaning of egg yolk deposit.

5.1.8 Removal of egg yolk deposit soaked in cleaning agents at different stirring conditions

Set of experiments were designed in order to study the effect of different stirring conditions on the cleaning of egg yolk deposit. Magnetic stirrer was used to provide low mixing while mixer impeller was used to provide high mixing to the cleaning solutions. After 120 minutes of experiment (Figure 5.3(a)), all samples soaked in water, sodium hydroxide solution, LAS solution and detergent solution stirred using mixing impeller give the highest deposit removal (8.89 $\pm$ 1, 13.58 $\pm$ 1, 12.82 $\pm$ 1, and 11.36 $\pm$ 1 %) followed by all samples soaked in all cleaning solutions stirred by magnetic stirrer (7.89 $\pm$ 1, 11.89 $\pm$ 1, 11.11 $\pm$ 1, and 10.52 $\pm$ 1 %) and all samples soaked in all cleaning solutions without mixing (6.98 $\pm$ 1, 10.81 $\pm$ 1, 9.81 $\pm$ 1, and 7.31 $\pm$ 1 %).

Stirring was used to provide mechanical forces to the cleaning of egg yolk deposit. Results from this set of experiment also suggest that mechanical forces play an important role to improve the cleaning of the egg yolk deposit by up to 55%. Samples soaked in detergent solution stirred using mixing impeller were found to have the highest deposit removal increase (55.4%). Without chemical action effect (samples soaked in water), stirring was found to increase the deposit removal by up to 38.33 %. Thus it indicates that mixer impeller was identified to provide higher deposit removal to

the egg yolk deposit than magnetic stirrer. Data obtained from these experiments suggest that mechanical forces help to raise the cleaning of egg yolk deposit.

5.1.9 Removal of egg yolk deposit soaked in cleaning solutions at different sample configurations

Experiment was designed to investigate the effect of different sample configurations on the cleaning of egg yolk deposit. After 120 minutes of experiment (figure 5.3(a) and 5.3(b)), all samples soaked in water, sodium hydroxide solution, LAS solution and detergent solution at vertical configuration (7.41 $\pm$ 1, 16.67 $\pm$ 1, 13.57 $\pm$ 1, 9.09 $\pm$ 1 %) give higher deposit removal than samples soaked in all cleaning solutions at horizontal configuration (6.98 $\pm$ 1, 10.81 $\pm$ 1, 9.81 $\pm$ 1 and 7.32 $\pm$ 1 %).

The difference in deposit removal between samples soaked in all cleaning agents tested at horizontal and samples soaked in all cleaning fluid tested at vertical configuration is up to 54 % with samples soaked in sodium hydroxide solution happen to give the highest difference between both configurations.

However, the difference in deposit removal between samples soaked in water (without chemical and physical action effect) at both configurations is only 6.16 %. Thus it can be concluded that different configurations does not really affect the deposit removal of egg yolk deposit.

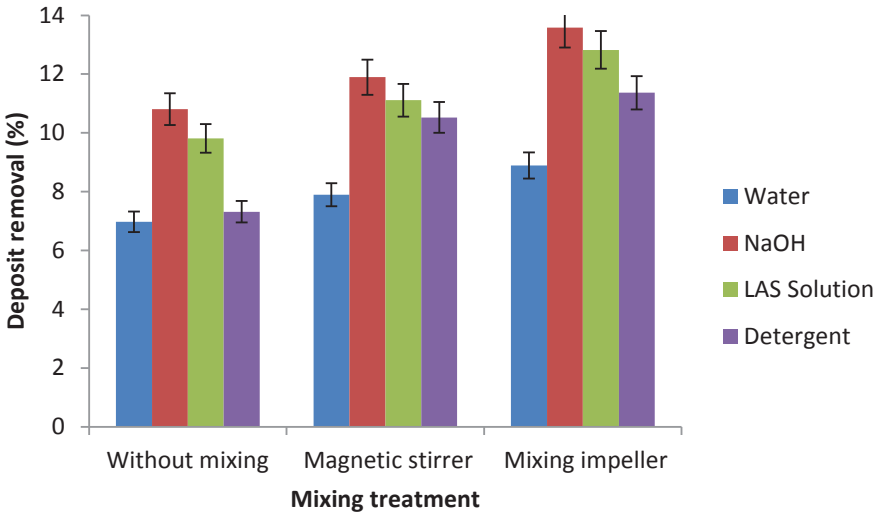


Figure 5.3 (a): Deposit removal of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at horizontal sample configuration at 3 different mixing treatments

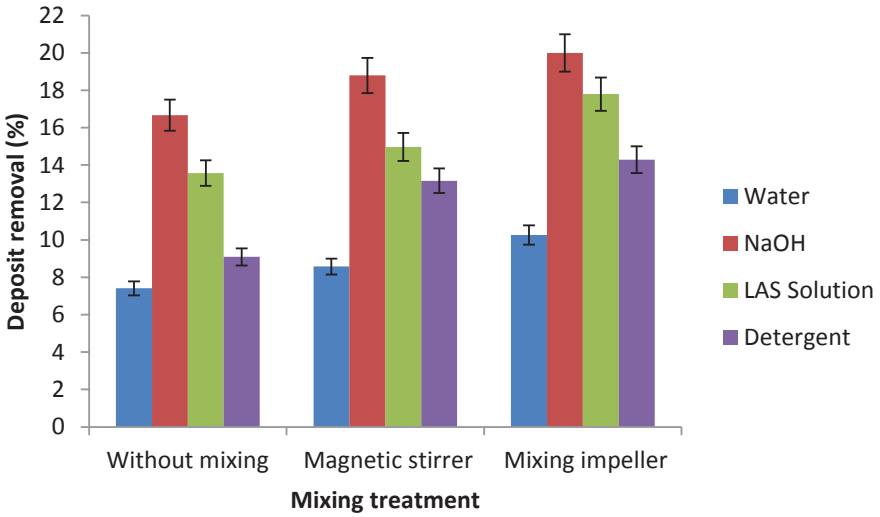


Figure 5.3 (b): Deposit removal of egg yolk deposit soaked in water, 1% w/v sodium hydroxide solution, 1% w/v LAS solution and 1% w/v detergent solution at vertical sample position at 3 different mixing treatments

5.2 Discussions

5.2.1 Moisture content and swelling performance of egg yolk deposits

It is well understood that moisture content and swelling of deposit is related to cleaning process. *Labuza* and *Hyman* (1998) reported that moisture content plays a key role in physical and chemical properties of food samples. Absorption of water into a food sample occurs when a low hydrated food sample is in contact with high moisture or liquid environments, which increases the volume of the food sample (*Perez-Mohedano et al*., 2016) and leads to an increase in moisture content and swelling of the food sample. A research by *Visser* (1995) reported that surface hydration and steric hindrance play an important role in reducing the adhesive force by preventing the close approach of fouling material and surface. *Liu et al* (2002) found that hydration of tomato paste deposit in cleaning fluid helps to weaken the adhesive force strength between surface and deposit which consequently ease the deposit removal. A cleaning research by *Fryer et al* (2006) also found that hydration of the deposit-surface interface was a key mechanism for deposit removal (Cole, 2011). It can be seen from the results of this present study that hydration increasing the moisture content and the swelling performance of egg yolk deposit leads to increase in deposit removal of egg yolk deposit.

Generally, fluid acts as a transport required to bring the chemical to the fouling material. Swelling happens when reaction occurred after the deposit is being contacted with the chemical, which leads to deformation of deposit structure (*Aziz*, 2008). A research by *Liu et al* (2006) using sodium hydroxide solution at different concentrations as hydration fluid suggested that the whey protein concentrate deposit swells after being exposed to

sodium hydroxide. Saikhwan et al (2010) also found that pH of hydration fluid controlled the swelling of whey protein gels. This agrees with the finding for hydration of egg yolk deposit in this present research that swelling performance of egg yolk deposit is increased by up to 40% after being soaked in alkaline cleaning solution for 120 minutes. On the other hand, *Christian and Fryer* (2003) suggested that the fluid motion is needed to bring the chemical to the deposit surface interface and help to in increase the mass transfer of the cleaning chemical into the deposit which agrees with the current study results that stirring of cleaning solutions using magnetic stirrer and mixing impeller may help to increase the moisture content and swelling performance of egg yolk deposit by up to 25% and 32% respectively.

5.2.2 The effect of chemical action on cleaning of egg yolk deposits

As a type 3 deposit, egg yolk need chemical action to be removed from its surface rather than water alone (*Fryer* and *Asteriadou*, 2009). *Plett* (1985) has reported that cleaning with detergent gives a maximum cleaning rate and diffusion of the chemical in to the deposit during cleaning will transform the deposit in to a removable form, through saponification of fats, emulsification and melting (*Fryer* and *Asteriadou*, 2009). Plett (1985) has also identified three different mechanisms that involved in chemical actions during cleaning i.e;

i) *Dissolution*; dissolving of food material by cleaning fluid used such as dissolving mineral salts in acids, proteins in alkali and sugars in water. Mass transfer to and from surface, dissolution kinetics and heat transfer (if thermal system involved) were believed to be the key player in this mechanism.

ii) Bulk cohesive failure; chemical actions are involved in the breakdown of the cohesive force between the molecules of the fouling material which abandon the deposit on the surface.

iii) *Surface adhesive failure*; chemical actions are involved in the breakdown of the adhesive force between the fouling materials and the surface which lead to the removal of the fouling materials.

In this research, egg yolk deposit was found to be removed by the bulk cohesive failure mechanism. During the cleaning process, proteins in egg yolk swell when on contact with alkaline cleaning solution which act to break the cohesive force between the deposit particles and gradually remove the top layer of the deposit, leaving the bottom layer of the deposit on the surface.

It is crucial to choose the most efficient cleaning agent for economic cleaning. Alkaline cleaning agents are usually used for protein and fat based soils. Reaction occurs when alkali are in contact with the deposit. Hydrolysis of peptide bonds and solubilisation of protein will occur to break the protein structure while saponification takes place to break the fat structure (Aziz, 2008). Sodium hydroxide solution, LAS solution and a commercial detergent solution (Arial) were chosen to be used as alkaline cleaning agents in these studies because it is well understood that 60% of the egg yolk composition is made up by proteins and fats. This agrees with the results shown in section 5.3.1 which found that sodium hydroxide solution, LAS solution and commercial detergent solution give higher deposit removal compares to water; with sodium hydroxide solution was found to have the highest deposit removal.

5.2.3 The effect of mechanical action on cleaning of egg yolk deposits

For a type 3 soil cleaning, the mechanical force created by water flow alone is not sufficient enough to remove the deposits. Chemical actions are needed to remove the deposits from the surface (*Christian* and *Fryer*, 2003). However, the fluid motion is needed to bring the chemical to the deposit surface interface, to provide shear to detach the deposit from the surface, and to carry the detached soil out of the system (*Christian* and *Fryer*, 2003). There are many researchers who have reported the mechanical effects of fluid flow in cleaning process of different fouling materials using several methods. *Timperley* and *Smeulders* (1988); *De Goederen et al* (1989); *Bird* and *Fryer* (1991); and *Othman et al* (2010) reported that increase in flow rates of cleaning fluid led to decrease in cleaning time of deposit. *Christian* (2004) also stated that higher flow rates provide higher shear stress on the deposit and increase in flow speed leads to decrease in boundary layer which helps to promote the removal of the food sample deposit. In this present study, stirring using magnetic stirrer and mixing impeller were found to create mechanical force (fluid flow) which helps to improve the cleaning of egg yolk deposit. As we could see from the results (figure 3.3 (a) and (b)), stirring was found to give higher deposit removal compares to samples soaked in the cleaning solutions without stirring.

5.2.4 Chemical action vs mechanical action

As reported earlier, cleaning of type 3 deposits is known to be the combination of chemical and mechanical action. *Perez-Mohedano et al* (2016) conducted studies on swelling and hydration of egg yolk deposit using scanning fluid dynamic gauge and gravimetric tests. Gravimetric tests were conducted to measure the mass of deposit

(moisture content) throughout the experiment. Results from the tests show that the mass of egg yolk deposit increased when the temperature and pH of hydration fluid increased. At the lowest pH tested (9.5), temperatures show no significant difference in deposit mass. There is no mechanical action applied in these experiments because gravimetric tests were conducted without any gauging action occurring. Therefore, they came up with a conclusion that pH (chemical action) has greater effect than temperature on the mass of deposit (moisture uptake) during hydration process within the levels studied.

In this current research, egg yolk samples soaked in sodium hydroxide solution give higher moisture content compares to samples soaked in water (figure 5.1 (a)). The increase in moisture content is found to be 77.92%. On the other hand, samples soaked in water stirred by mixing impeller have higher moisture content compares to samples soaked in water without stirring (figure 5.1 (a) and 5.1 (f)). The increase in moisture content is found to be 39.3%. From these results, the effect of chemical action was known to be greater than the effect of mechanical action on the moisture content of egg yolk deposit.

5.2.5 Swelling performance of egg yolk deposit

Liu et al (2006) stated that swelling occurs when whey protein concentration (WPC) deposit soaked in sodium hydroxide solution at different concentration. *Christian* and *Fryer* (2003) also reported that swelling of WPC deposit during intermediate section of WPC cleaning using plate heat exchanger (PHE) was found to be the effect of chemical action. *Perez-Mohedano et al* (2016) stated that increase in temperature and pH of

hydration fluid led to increase in swelling of egg yolk deposit. However, it was found that the effect pH (chemical action) was greater than temperature. *Gordon et al*. (2012) found that chemical action (protease) and mechanical action (shear stress) suppress the swelling of egg yolk deposit layer soaked in buffer solution and reduced the final deposit thickness through deposit removal.

In the current study on swelling performance of egg yolk, samples soaked in sodium hydroxide solution have higher swelling performance compares to samples soaked in water (figure 3.2 (a)). The swelling performance is found to be increased by 87.74%. Samples soaked in water stirred by mixing impeller show higher swelling performance compares to samples soaked in water without stirring (figure 3.2 (a) and 3.2 (f)). The swelling performance is raised by 41.43%. It is clearly suggested by these results that chemical action has greater effect on the swelling performance of egg yolk deposit than mechanical action.

5.2.6 Removal of egg yolk deposit

Cleaning of egg yolk deposit requires both chemical and mechanical action. Plett (1985) found that introducing detergent into cleaning gives a maximum cleaning rate and increase in flow rate may reduce the cleaning time. Othman *et al* (2010) carried out studies to investigate the cleaning of sweet condensed milk (SCM) using a flow cell suggested that increasing the flow rate from 0.25 to 0.5 $ms^{-}1$ reduced the cleaning time of SCM. However, they also found that increasing the concentration of sodium hydroxide solution from 0.5 to 1.5% does not really affect the cleaning time of SCM. Therefore, they came up with a conclusion that the optimum concentration of sodium

hydroxide solution for cleaning is 0.5%. *Christian* (2004) and *Aziz* (2008) conducted studies on cleaning of type 3 deposits i.e. whey protein concentration (WPC) and egg albumin gel deposit to investigate the effect of flow rate and temperature of water on cleaning. They found that none of the deposit was removed by water at temperatures (30 – 70 ^{o}C) and flow rates (0.7 – 2.3 lmin^{-1}) tested. *Aziz* (2008) also suggested that increasing the flow rate of cleaning fluid does not really affect the cleaning time of albumin gel deposit at all sodium hydroxide concentration.

Perez-Mohedano et al (2015) did studies to develop a swelling-removal model for the hydration of egg yolk deposit. Scanning fluid dynamic gauge was used in this studies to provide shear stress (mechanical action), and protease was used to provide chemical action. Three tests were conducted with different conditions. Test 1 was conducted without protease and external shear stress, test 2 was carried out with protease but without external shear stress and test 3 was conducted with protease and external shear stress. Results for these studies were shown in terms of thickness of deposit. Removal of deposit was shown by decrease in deposit thickness. No deposit removal was shown in test 1. Test 2 shows that deposit thickness start to decrease after 30 minutes of experiment and the deposit layer is completely removed after 100 minutes of experiments while test 3 shows that deposit removal occurs after 15 minutes of experiment and the deposit layer is completely removed after 30 minutes of experiment. Both chemical and mechanical action plays important role in deposit removal. Adding shear stress into the system reduces the cleaning time by approximately 70%. Thus, it can be concluded that mechanical action plays a bigger role than chemical action in this cleaning experiment.

Gordon et al (2012) carried out research to study enzyme-based cleaning of gelatin and egg yolk using scanning fluid dynamic gauge. Protease was used to provide chemical action while scanning fluid dynamic gauge was used to provide shear stress (mechanical action). Results were shown in terms of deposit thickness. Sets of experiments were designed to investigate the effect of protease and shear stress on the cleaning of egg yolk deposit. Figure shows that egg yolk sample soaked in buffer swells constantly until it reaches equilibrium after 100 minutes of experiment. When protease was introduced into the system, deposit swelling was interrupted after 40 minutes of experiment and deposit removal starts after 60 minutes of experiment and totally removed after 140 minutes of experiment. Figure 5.5b shows the doubling effect of shear stress applied by the gauging fluid from 20 to 40 Pa which reduced the cleaning time by 20%. Experiment with protease shows that the deposit is totally removed after 140 minutes while experiment with 20 and 40 Pa of shear stress show that the deposit was totally removed after 210 and 150 minutes. They also reported that shear stress applied should be higher than 10 Pa in order to remove the deposit. Thus it can be concluded that the effect of chemical action is greater than the effect of mechanical action in this set of experiments.

As for this present study, egg yolk samples soaked in sodium hydroxide were found to give higher deposit removal compares to samples soaked in water (figure 5.3 (a)). The deposit removal of egg yolk was increased by 54.87%. On the other hand, samples soaked in water stirred using mixing impeller have higher deposit removal compares to samples soaked in water without stirring (figure 5.3 (a)). The deposit removal was found to be increased by 27.36%. However, the egg yolk deposit is not completely removed

after 120 minutes of experiment and results show that there is no sign of deposit thickness reduction because swelling and deposit removal occurs simultaneously in this set of experiments.

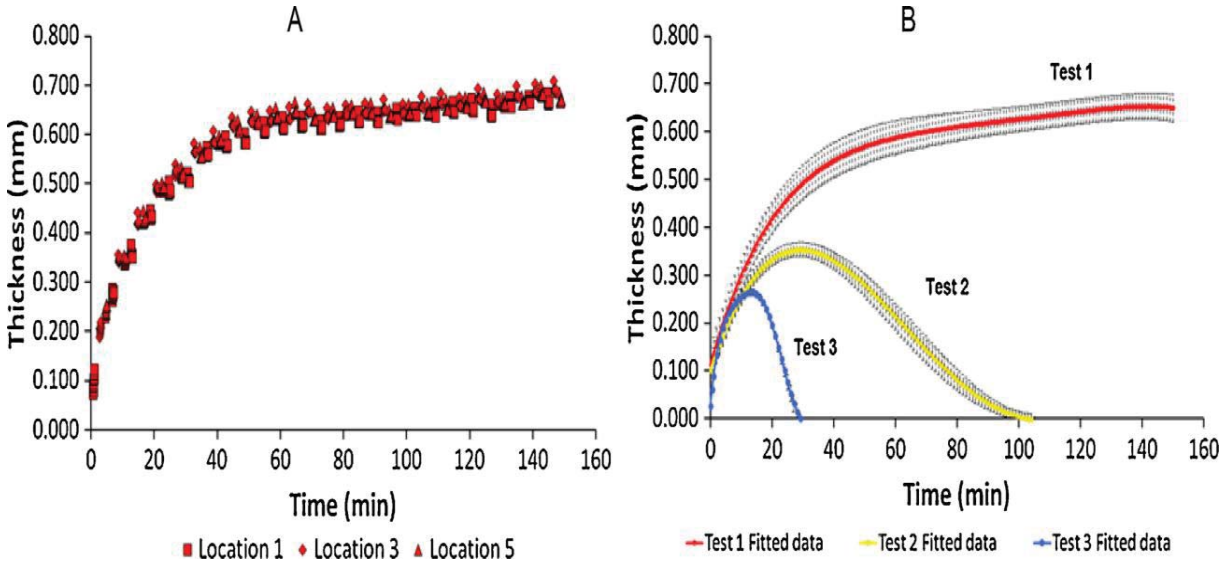


Figure 5.4: **Raw data values for 3 different locations on the same tile in a single test 1 experiment. B – Averaged fitted experimental results for the three experimental tests considered (Perez-Mohedano et al., 2015)**

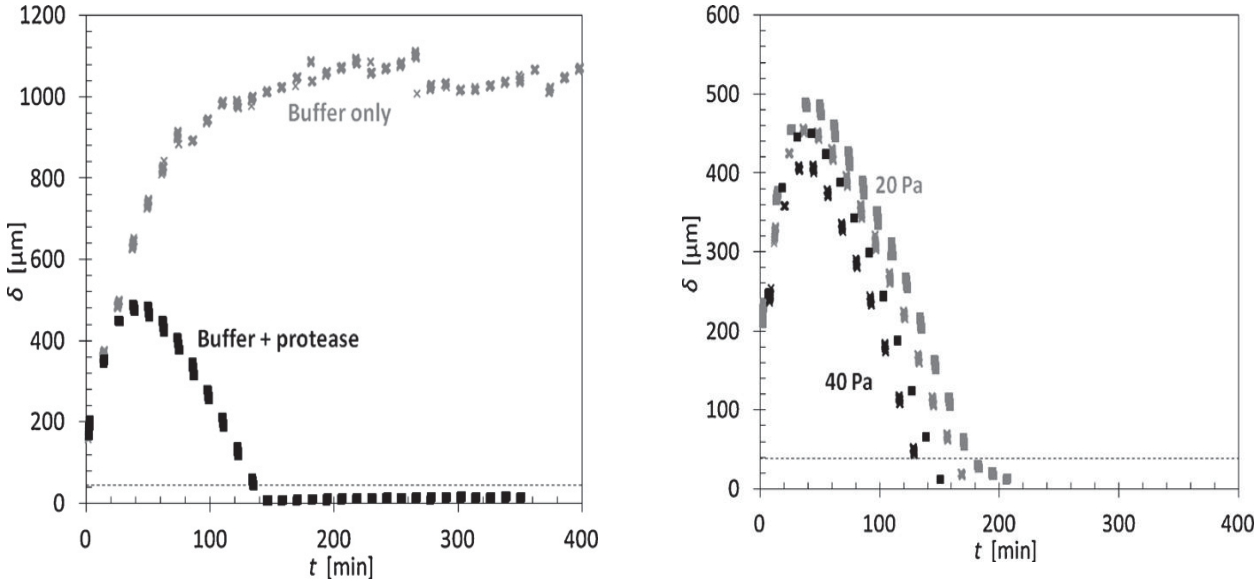


Figure 5.5: A) Comparison of swelling behaviour in pH 10.8 buffer solutions and buffered protease solution at 40 ◦C. Horizontal dashed line denotes the initial dry thickness of the protein layer. No removal was observed in areas that were not studied with the gauge. B) Effect of increasing the shear stress exerted by the gauging fluid from 20 Pa (grey data points) to 40 Pa (black) on time taken to clean the egg yolk deposit. Two points on the surface of the egg yolk layer, denoted by squares and crosses, were measured for each shear stress. (Gordon et al., 2012)

5.2.7 Novelty and innovations

The hydration experiment set up was specifically designed to investigate the effect of mechanical and chemical action on the swelling and the cleaning of egg yolk deposit at room temperature. Magnetic stirrer and mixing impeller are used to provide mechanical force while sodium hydroxide, LAS solution and a commercial detergent are used to provide chemical action for the experiment carried out. Egg yolk powder is used to prepare the deposit sample as it contains high fat content. Swelling performance and deposit removal percentage are calculated in order to obtain the results from the experiments. The swelling and the cleaning of egg deposits are controlled by chemical and mechanical action. However, chemical action has greater effect on the swelling and

the cleaning of egg yolk deposits compares to mechanical action. No complete removal was shown after 2 hours of experiment in all cases.

5.3 Conclusions

This hydration experiment can be used to study the cleaning of egg yolk deposits in terms of moisture content, swelling performance and deposit removal during hydration. It was found that physical action and chemical action play important role in the swelling and deposit removal of egg yolk deposits.

The Introduction of sodium hydroxide solution, LAS solution and commercial detergent solution provides chemical action to the hydration of egg yolk deposit and increase the moisture content of deposits. Sodium hydroxide gives the highest moisture content increase followed by LAS solution and commercial detergent solution.

Stirring provides mechanical action to the system and increase the moisture content of egg yolk deposit (without chemical action) with mixing impeller gives higher moisture content increase than magnetic stirrer.

Different sample configuration does not really affect the moisture content of egg yolk deposits as only a slight different in moisture content is found between samples soaked at horizontal configuration and those samples soaked at vertical configuration.

It was understood that swelling is related to the moisture content of the deposit. Thus, chemical action also plays a key role in swelling performance of egg yolk deposits. All samples soaked in all cleaning chemical tested increase the swelling performance compares to samples soaked in water with sample soaked in sodium hydroxide have the highest swelling performance.

Magnetic stirrer and mixing impeller provides physical action to the hydration system and was identified to increase the swelling performance of egg yolk deposits by up to 38.29 % without chemical action.

It is found that swelling performance of egg yolk deposits was affected by different sample configurations. The difference in swelling performance between samples soaked at both configurations without chemical and mechanical is 18.06 %. Surprisingly, the difference in swelling performance between samples soaked at both configurations with chemical action but without mechanical action is from 6.95 to 11.01 %. Thus, it can be concluded that the effect of different sample configurations is not well understood.

Without physical action, sodium hydroxide solution is found to be the best chemical used to clean the deposit with 55.01 % increase in deposit removal compares to deposit soaked in water, LAS solution gives 40.75 % deposit removal increase and detergent solution helps to raise the deposit removal by 5.02 %. This agrees with the previous workers findings that chemical action affect the removal of deposits.

Stirring using magnetic stirrer and mixing impeller is found to affect the deposit removal of egg yolk deposit. This physical action is known to increase the deposit removal by up to 38.33 % without chemical action effect. Therefore, this is aligned with the previous studies that mechanical/physical action plays an important role in the cleaning of food fouling deposit.

The difference in deposit removal between samples soaked at both configurations without chemical and physical action effect is only 6.16 %. Thus it can be concluded

that different configurations does not really affect the deposit removal of egg yolk deposit.

Swelling and deposit removal occurs simultaneously in some cases. Mechanical and chemical action affects the swelling performance and removal of deposits. Increase in swelling performance leads to increase in removal of egg yolk deposits.

CHAPTER 6: CLEANING OF EGG YOLK DEPOSITS AT LABORATORY SCALE

This chapter will focus on cleaning of egg yolk deposits at laboratory scale using cleaning rig (flow cell rig) and determination of force required to remove egg yolk deposits using micromanipulation technique. The effect of different cleaning agent, different cleaning temperature and chemical concentration on cleaning of egg yolk deposits were studied and the effect of hydration temperature, hydration time and chemical concentration of hydrating solution on the adhesive and cohesive strength of egg yolk deposits were investigated.

6.1 Results

6.1.1 Heat transfer coefficient, U profile

Heat transfer coefficient (U) profile is used in this research in order to study the cleaning progress of the egg yolk deposit. This (U) profile can be used as a method to measure the cleaning time. The insulating deposit layer is removed gradually throughout the experiment which increases the heat transfer coefficient value until it reaches plateau which indicates the clean stage. The calculations for heat transfer coefficient are discussed in section 2.21. In this set of experiments, cleaning agents (water, sodium hydroxide solution, LAS solution) were rinsed at different concentration (1 and 5 % v/w) and temperatures 40, 60, and 80^{o}C through the cleaning rig in order to study cleaning of egg yolk deposit using water as cleaning fluid. All experiments were conducted at 0.5 ms^{-1} flow velocity. The cleaning of egg yolk deposit can be characterized by heat transfer coefficient (U) profile. According to Goode (2012), this U profile can be divided into 3 phases;

a) Lag phase; where no deposit removal occurs,

b) Removal phase; where deposit removal takes place,

c) Constant phase; where all the deposit has been removed from the surface and no further deposit removal occurs.

Figure 6.1 show that there is no removal phase and constant phase takes place in this experiment. Only lag phase was shown in this U profile. No complete deposit removal after 4 hours of experiment. This indicates that egg yolk deposit cannot be cleaned by water alone even at high temperature.

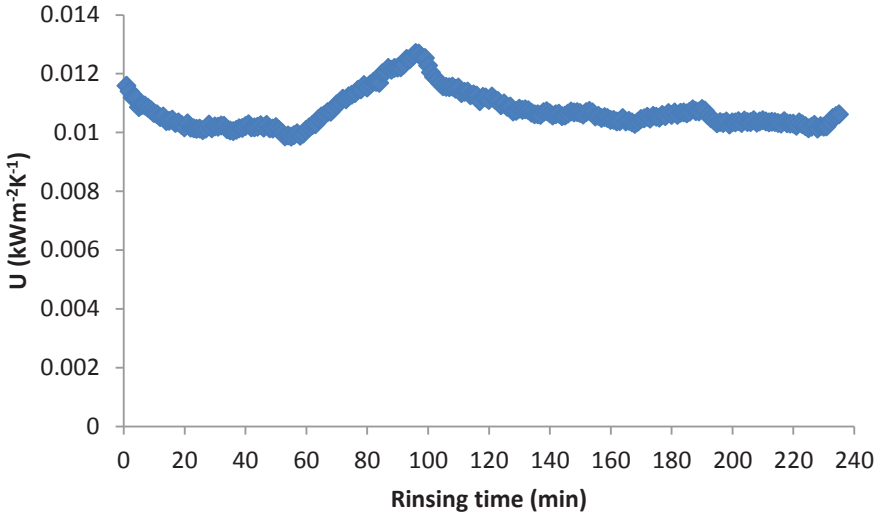


Figure 6.1: U profile for egg yolk deposit cleaning using water at 80^{o}C and 0.5ms^{-1}

Figure 6.2 also shows no removal and constant phase occurred. Only lag phase was shown on the profile. No complete deposit removal was achieved after 2 hours experiment. This indicates that cleaning with sodium hydroxide 1 % w/v at 40^{o}C and 0.5 ms-1 is not sufficient enough to clean all the egg yolk deposit from the stainless steel surface. Figure 6.3 shows complete removal of egg yolk deposit after 175 minutes of experiment. Lag phase takes place for 6000 s during the experiment before removal

phase starts at about 6000 s and constant phase occurred after 10000 s of experiment. This suggests that cleaning of egg yolk deposit using sodium hydroxide 1 % w/v at 60^{o}C and 0.5 ms-1 is sufficient to clean all the deposit from the surface. Figure 6.4 illustrates the cleaning of egg yolk with sodium hydroxide 1 % w/v at 80^{o}C and 0.5 ms^{-1}. The first 600 s of the U profile represents lag phase and removal phase occurs from 600 s until it reach constant phase at 2000 s. This U profile also shows that complete deposit removal happens after 33 minutes of experiment, thus indicates that this cleaning condition can be used to clean the egg yolk deposit effectively.

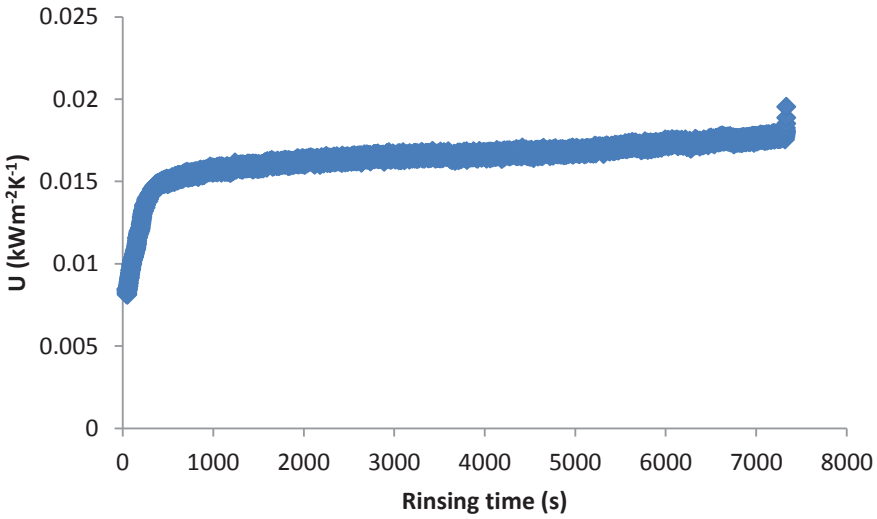


Figure 6.2: U profile for egg yolk deposit cleaning using 1 % w/v sodium hydroxide at 40^{o}C and 0.5 ms^{-1}

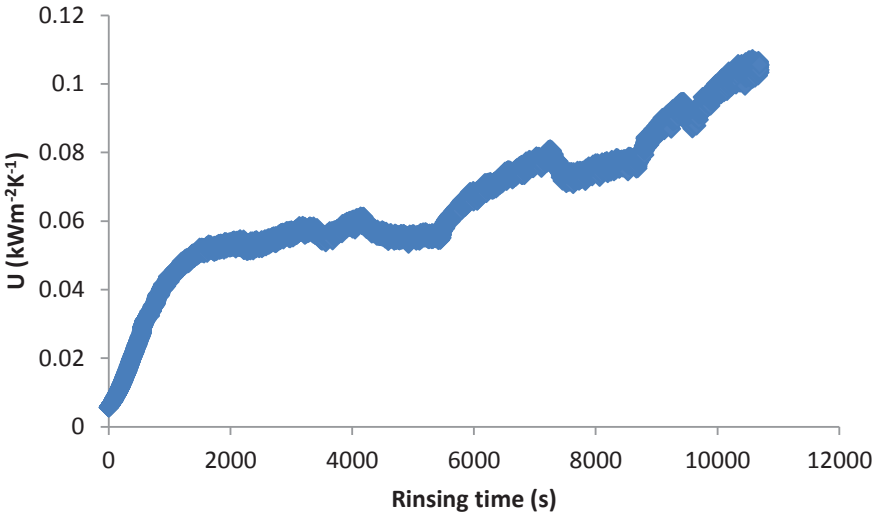


Figure 6.3: U profile for egg yolk deposit cleaning using 1 % w/v sodium hydroxide at 60°C and 0.5 ms^{-1}

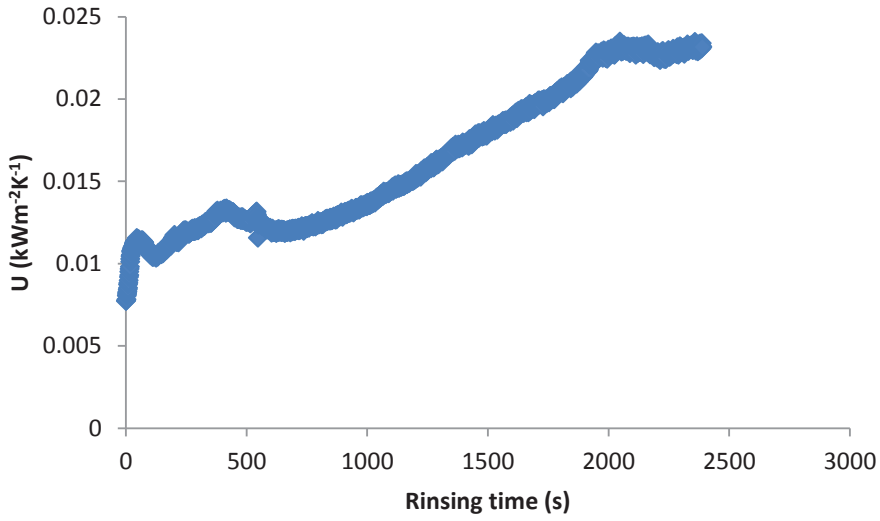


Figure 6.4: U profile for egg yolk deposit cleaning using 1 % w/v sodium hydroxide at 80°C and 0.5 ms^{-1}

Figure 6.5 shows U profile for egg yolk deposit cleaning using 5 % w/v sodium hydroxide at 40°C and 0.5 ms^{-1}. It was found that no complete deposit removal was shown after 2 hours of experiment. There is no removal phase and constant phase on U profile. Only lag phase was shown on the profile. Thus, it suggests that this condition is not sufficient enough to clean the egg yolk deposit from the surface. Figure 6.6 illustrates U profile for samples cleaned with 5 % w/v sodium hydroxide at 60°C and 0.5 ms^{-1}. Lag phase represents the first 2600 s of experiments while removal phase starts at about 2600 s and reaches constant phase at 4500 s. complete deposit removal was achieved after 75 minutes of experiment. Thus, it shows that this cleaning condition is sufficient to remove the egg yolk deposit from the surface. Figure 6.7 represents U profiles for egg yolk samples rinsed with 5 % w/v sodium hydroxide at 80°C and 0.5 ms^{-1}. The first 600 s of experiment was identified as lag phase while removal phase occurs after 800 s and deposit removal stops at 1300 s. All deposit was totally removed after 22 minutes of experiment. Therefore, it indicates that this cleaning condition can be used to clean egg yolk deposit from stainless steel surface effectively.

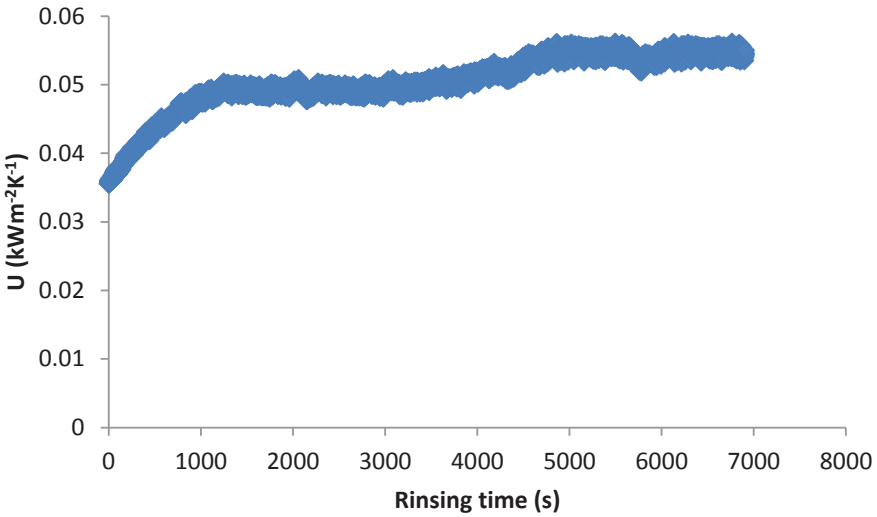


Figure 6.5: U profile for egg yolk deposit cleaning using 5 % w/v sodium hydroxide at 40°C and 0.5 ms^{-1}

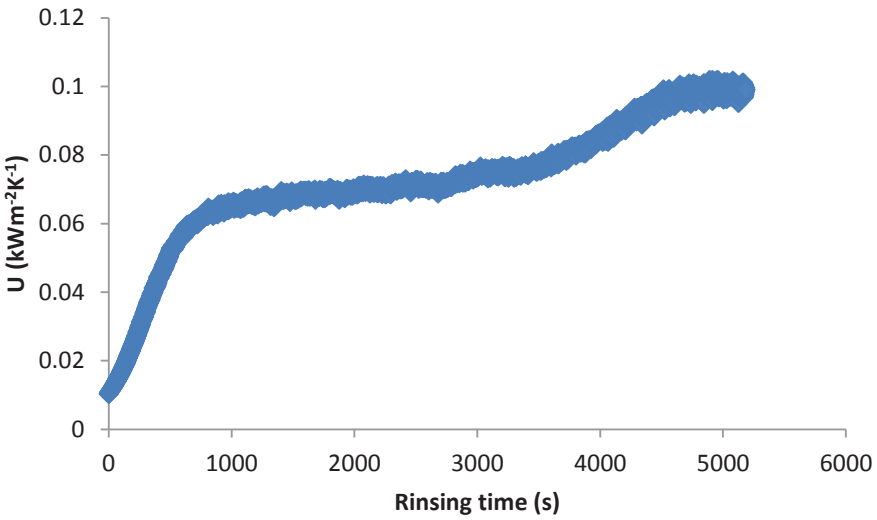


Figure 6.6: U profile for egg yolk deposit cleaning using 5 % w/v sodium hydroxide at 60°C and 0.5 ms^{-1}

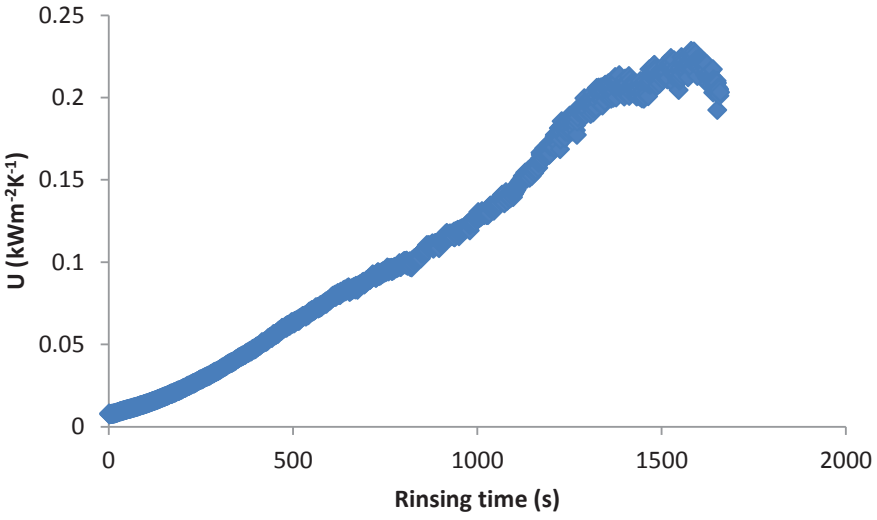


Figure 6.7: U profile for egg yolk deposit cleaning using 5 % w/v sodium hydroxide at 80°C and 0.5 ms^{-1}

Figure 6.8 shows U profile for cleaning of egg yolk deposit using 1 % LAS solution at 40^{o}C and 0.5 ms^{-1}. No removal phase or constant phase was found on this profile. Only lag phase was identified on this profile. Complete deposit removal was not achieved by this experiment. Thus, it suggests that this cleaning condition is not sufficient enough in order to clean egg yolk deposit from the stainless steel surface. Figure 6.9 illustrates U profile for egg yolk deposit cleaning with 1 % LAS solution at 60^{o}C and 0.5ms^{-1}. The first 8000 s of the experiment represents the lag phase and removal phase starts at about 8000 s and reaches constant phase at about 10300 s. Deposit was completely removed after 172 minutes of experiment. Therefore it indicates that this cleaning condition is sufficient to clean egg yolk deposit on stainless steel surface. Figure 6.10 represents U profile for egg yolk deposit rinsed with 1 % LAS solution at 80^{o}C and 0.5 ms^{-1}. The first 1600 s is identified as lag phase and deposit removal starts to happen after 1600 s and constant phase occurs after 2800 s of experiment. Deposit was totally removed after 47 minutes of experiment. Thus this suggests that this condition is sufficient enough to clean the egg yolk deposit from stainless steel surface.

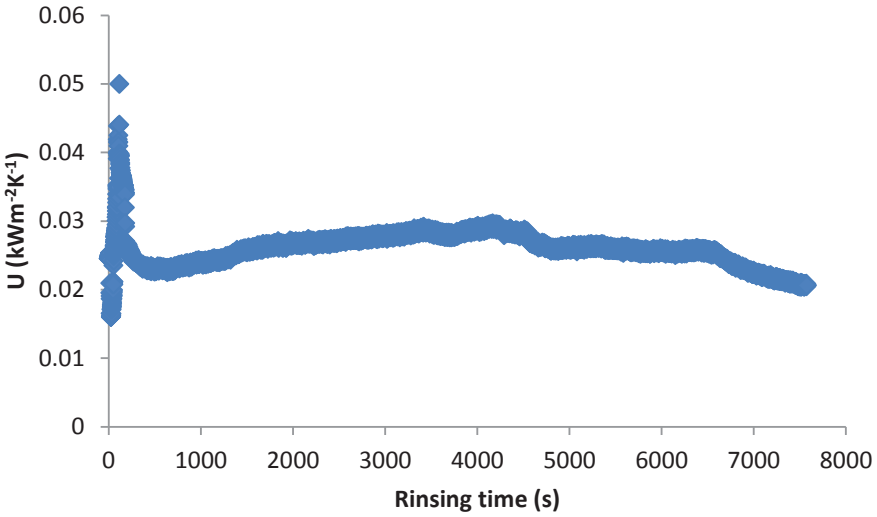


Figure 6.8: U profile for egg yolk deposit cleaning using 1 % w/v LAS solution at 40°C and 0.5 ms^{-1}

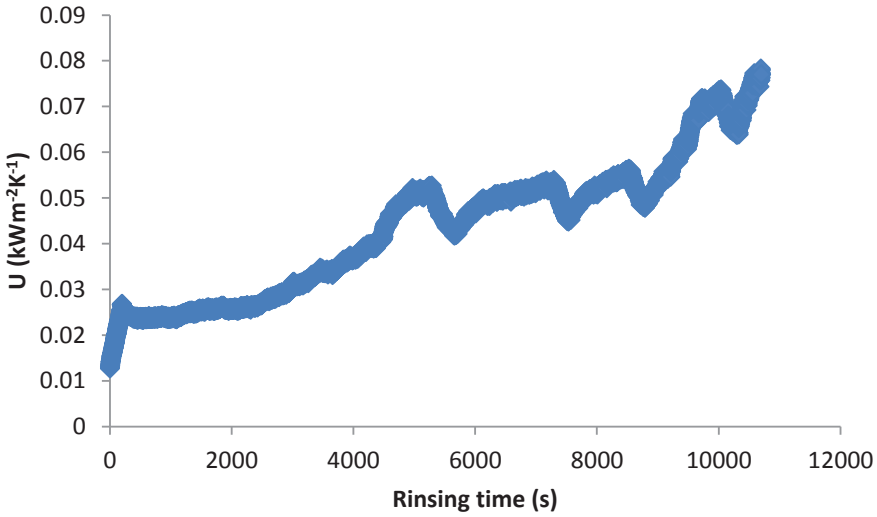


Figure 6.9: U profile for egg yolk deposit cleaning using 1 % w/v LAS solution at 60°C and 0.5 ms^{-1}

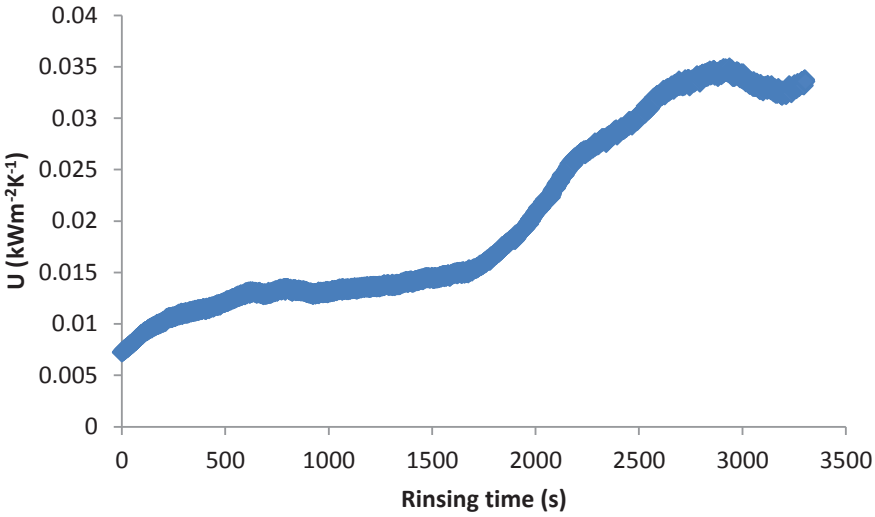


Figure 6.10: U profile for egg yolk deposit cleaning using 1 % w/v LAS solution at 80°C and 0.5 ms^{-1}

Figure 6.11 shows U profile for cleaning of egg yolk deposit with 5 % w/v LAS solution at 40°C and 0.5ms^{-1}. Only lag phase was found on this profile, no removal or constant phase. Complete removal of deposit was not achieved after 120 minutes of experiment. This suggests that this cleaning condition is not sufficient enough to clean egg yolk deposit fouled on stainless steel surface. Figure 6.12 illustrates U profile for egg yolk deposit cleaning using 5 % w/v LAS solution at 60°C and 0.5 ms^{-1}. Lag phase was found from 0 to 3300 s, followed by removal phase until 5300 s and constants phase starts after 5300s. Deposit was totally removed after of 88 minutes of experiment. Thus, it indicates that this cleaning condition can be used to clean the egg yolk deposit from stainless steel surface. Figure 6.13 represents U profile for cleaning of egg yolk deposit with 5 % w/v LAS solution at 80°C and 0.5 ms^{-1}. Lag phase was found on the profile from 0 to 1300s and removal phase occurs at 1300 s until 1900 s where constant phase was shown on the profile. All deposit was completely removed after 32 minutes of

experiment. Therefore, it suggests that this cleaning condition is sufficient enough to remove egg yolk deposit from stainless steel surface.

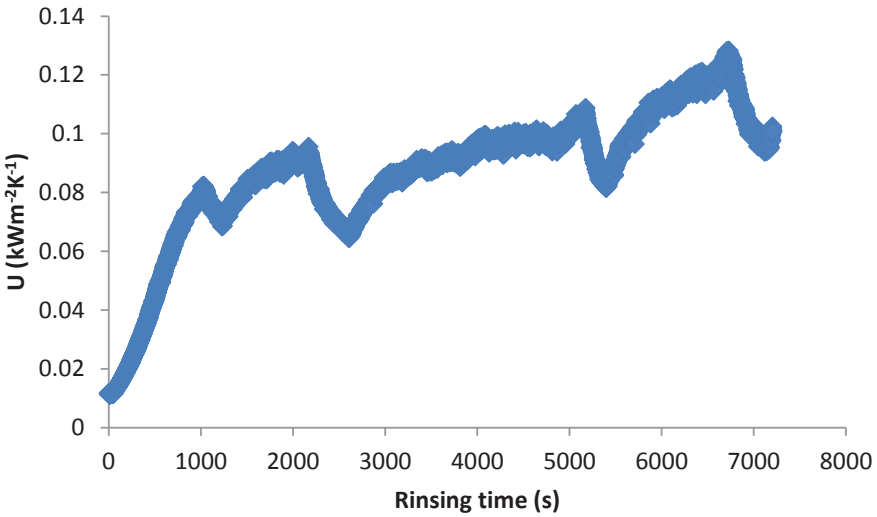


Figure 6.11: U profile for egg yolk deposit cleaning using 5 % w/v LAS solution at 40°C and 0.5 ms^{-1}

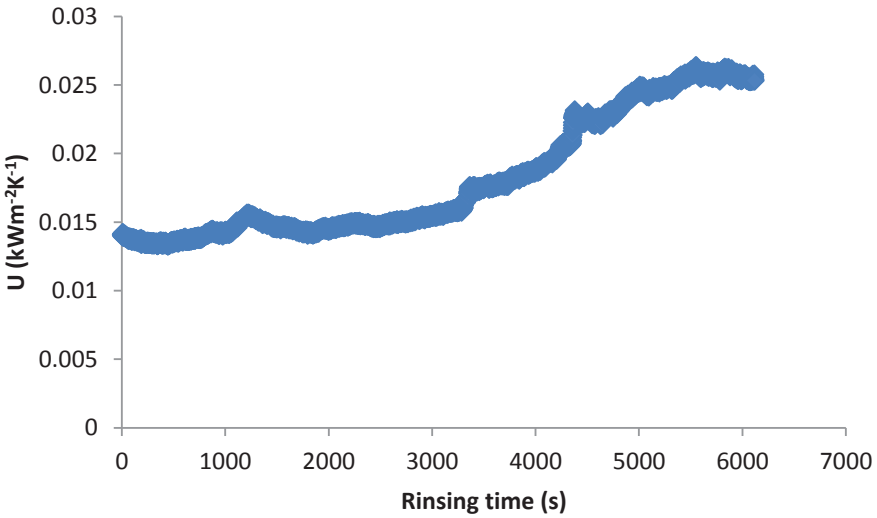


Figure 6.12: U profile for egg yolk deposit cleaning using 5 % w/v LAS solution at 60°C and 0.5 ms^{-1}

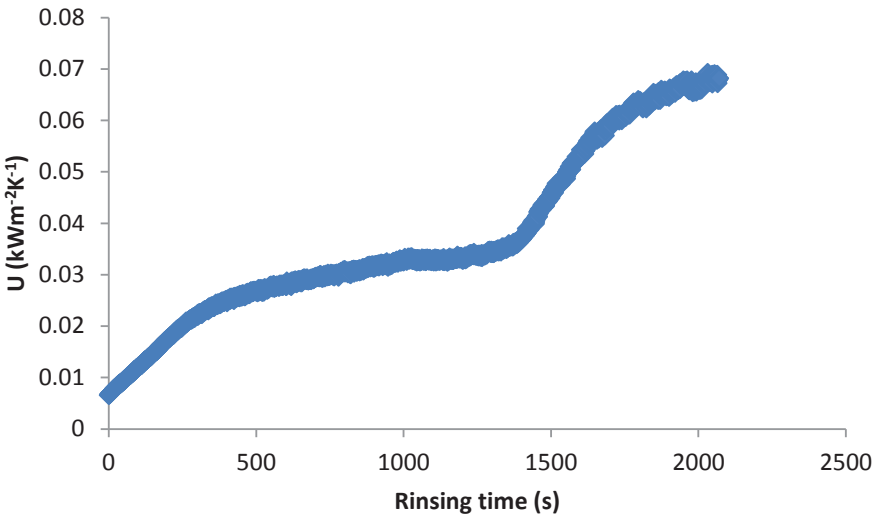


Figure 6.13: U profile for egg yolk deposit cleaning using 5 % w/v LAS solution at 80°C and 0.5 ms^{-1}

6.1.2 Deposit removal determined by image analysis

Apart from heat transfer coefficient profile, cleaning of egg yolk deposit also could be characterized by deposit removal profile from image analysis data. The deposit layer was seen to be removed gradually throughout the experiment until it was totally removed from the coupon (clean stage). The deposit removal was determined by the deposit layer area covered which was decreased throughout the experiment until it reaches zero (clean stage). The calculations for the deposit removal by image analysis are described in section 3.2.10. For all cleaning experiments at 40°C, final deposit left was calculated gravimetrically because it was difficult to determine the deposit left after experiment by image analysis technique. From the gravimetric results, it was found that 1 % w/v sodium hydroxide solution managed to clean only 7.53 % of egg yolk deposit while 5 % w/v sodium hydroxide solution was found to clean 22.37 % of egg yolk deposit. 1 % LAS solution was found to clean only 5.83 % of egg yolk deposit while 5 %

LAS solution managed to clean 16.93 % of egg deposit fouled on the stainless steel coupon.

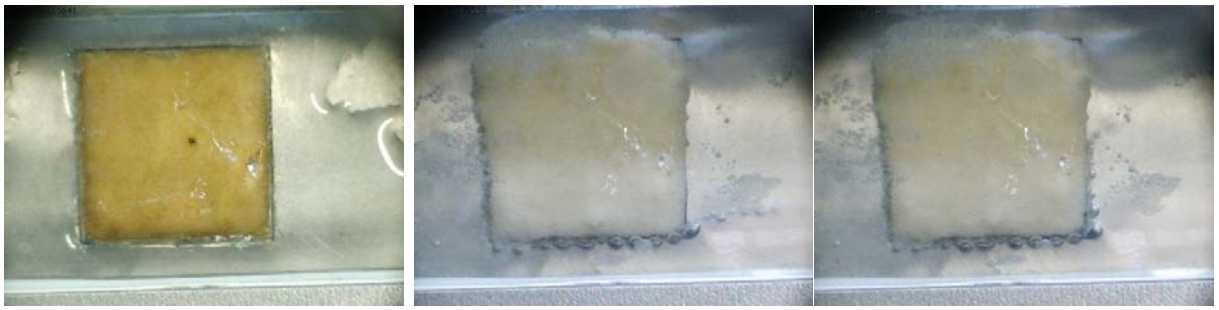


Figure 6.14: Image of egg yolk deposit at 1 min, 60 min and 120 minutes

Figure 6.15 represents deposit removal of egg yolk deposit samples rinsed with 1% w/v sodium hydroxide at 60^{o}C and 0.5 ms^{-1}. Removal phase was found to start after 100 minutes of experiment and deposit was completely removed after 180 minutes of experiment. Figure 6.16 illustrates cleaning of egg yolk deposit rinsed with 1 % w/v sodium hydroxide at 80^{o}C and 0.5 ms^{-1}. Deposit removal phase start to occur after 10 minutes of experiment and coupon was totally clean after 40 minutes of rinsing. Figure 6.17 shows cleaning of egg yolk deposit samples using 5 % w/v sodium hydroxide at 60^{o}C and 0.5 ms^{-1}. Deposit was found to be removed rapidly after 40 minutes of experiment and complete deposit removal was achieved after 80 minutes of experiment. Cleaning of egg yolk deposit by 5 % w/v sodium hydroxide at 80^{o}C and 0.5ms-1 was presented in figure 6.18. Data from image analysis suggests that removal of deposit occurs after 5 minutes of experiment with rapid deposit removal happens after 10 minutes of experiment. Deposit was totally removed after 25 minutes of cleaning.

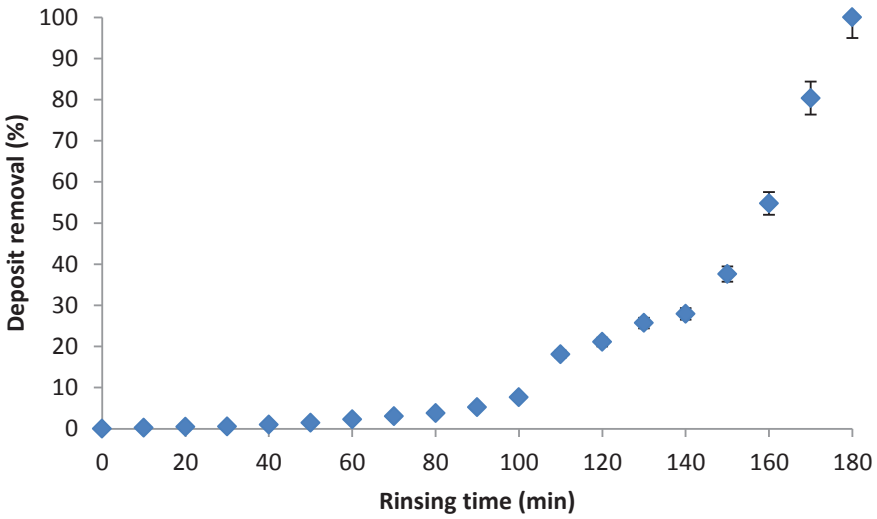


Figure 6.15: Deposit removal of egg yolk deposit rinsed with 1% w/v sodium hydroxide at 60°C and 0.5 ms^{-1}

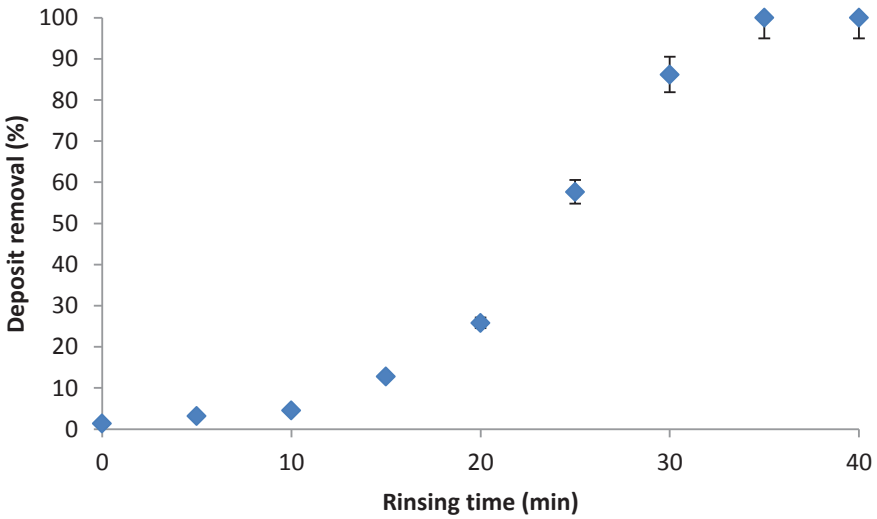


Figure 6.16: Deposit removal of egg yolk deposit rinsed with 1% w/v sodium hydroxide at 80°C and 0.5 ms^{-1}

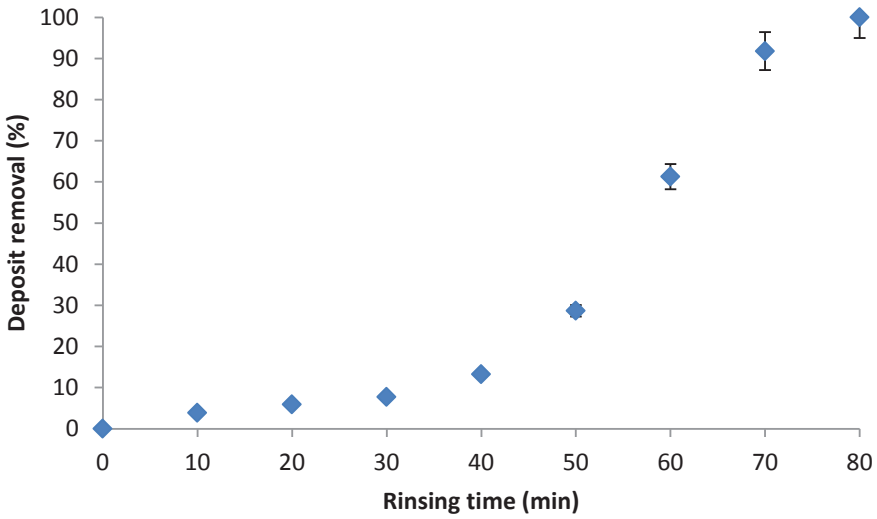


Figure 6.17: Deposit removal of egg yolk deposit rinsed with 5% w/v sodium hydroxide at 60°C and 0.5 ms^{-1}

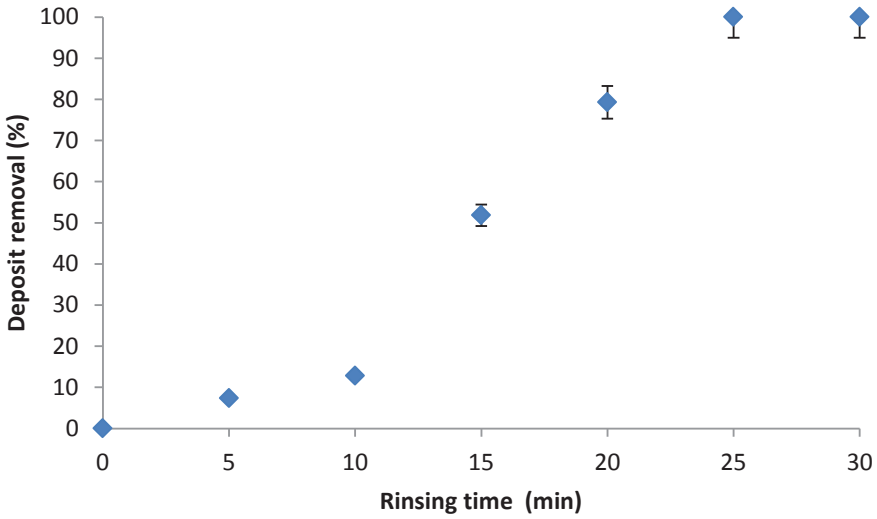


Figure 6.18: Deposit removal of egg yolk deposit rinsed with 5% w/v sodium hydroxide at 80°C and 0.5 ms^{-1}

Figure 6.19 shows cleaning of egg yolk deposit by 1 % w/v LAS solution at 60^{o}C and 0.5 ms^{-1}. It was found that removal start to occur at 80 minutes of experiment and rapid deposit removal happens after 135 minutes of experiment. Complete deposit removal was achieved after 180 minutes of experiment. Cleaning of egg yolk deposit by 1 % w/v LAS solution at 80^{o}C and 0.5 ms^{-1} was presented in figure 6.20. Data from image analysis suggests that removal occurs after 10 minutes of experiment and deposit removal increased rapidly after 20 minutes of experiment. Deposit removal stops after 50 minutes of rinsing and deposit was totally removed. Figure 6.21 illustrates cleaning of egg yolk deposit rinsed with 5 % w/v LAS solution at 60^{o}C and 0.5 ms^{-1}. Result from this experiment indicates that removal starts to occur after 10 minutes of experiment and rapid removal increment happens after 50 minutes of experiment. Deposit was completely removed after 90 minutes of experiment. Figure 6.22 represents cleaning of egg yolk deposit rinsed with 5 % w/v LAS solution at 80^{o}C and 0.5 ms^{-1}. Deposit removal starts after 5 minutes of experiment and increased rapidly after 20 minutes of experiment. Stainless steel surface was clean after 35 minutes of rinsing.

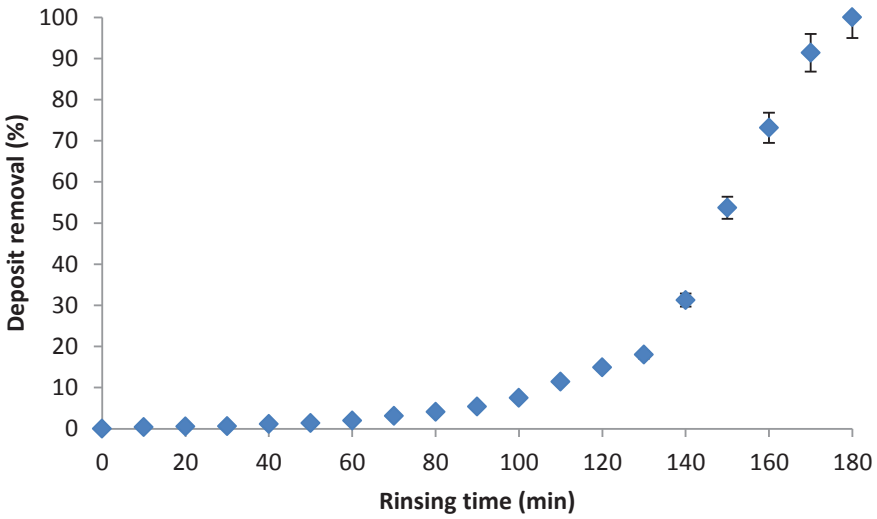


Figure 6.19: Deposit removal of egg yolk deposit rinsed with 1% w/v LAS solution at 60°C and 0.5 ms^{-1}

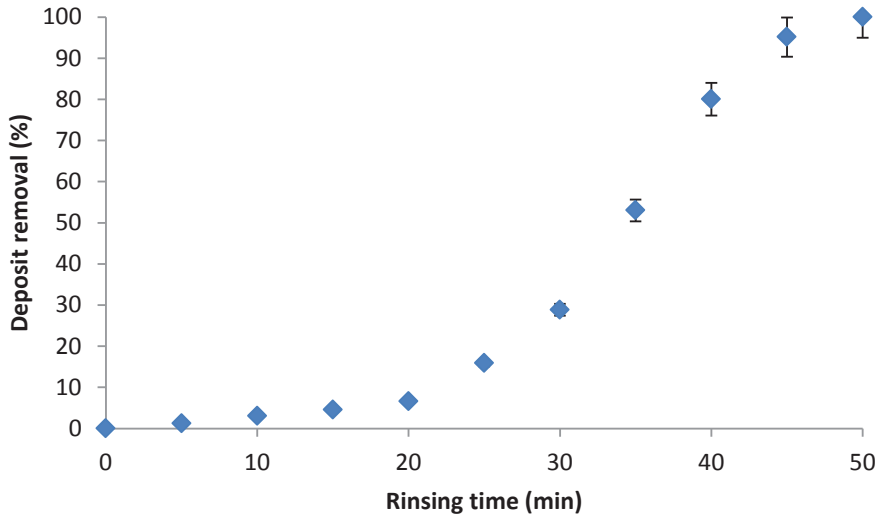


Figure 6.20: Deposit removal of egg yolk deposit rinsed with 1% w/v LAS solution at 80°C and 0.5 ms^{-1}

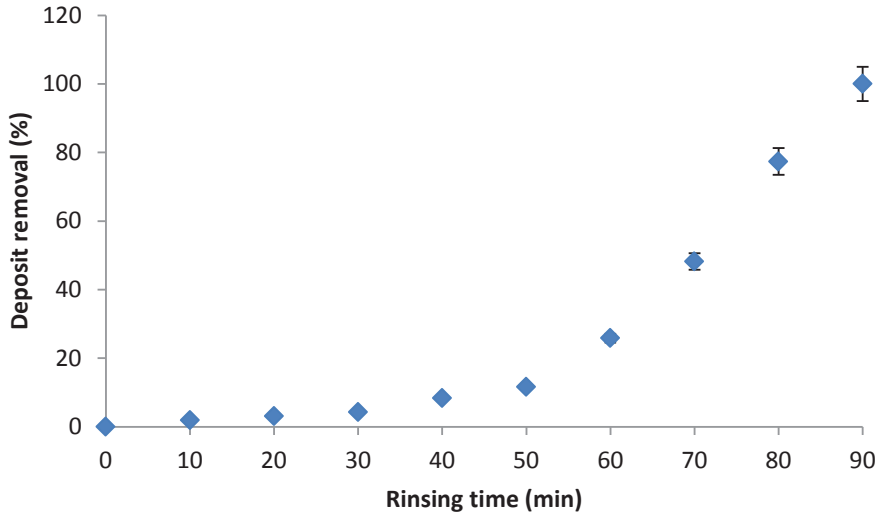


Figure 6.21: Deposit removal of egg yolk deposit rinsed with 5% w/v LAS solution at 60°C and 0.5 ms^{-1}

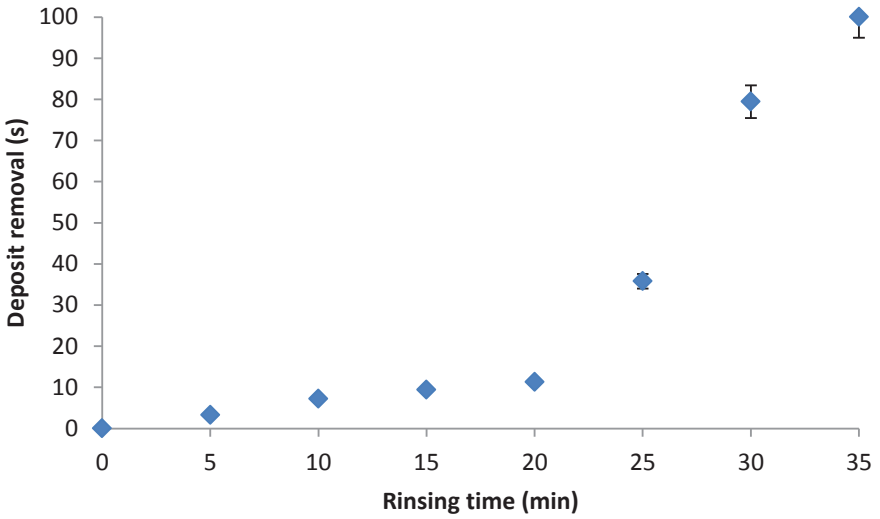


Figure 6.22: Deposit removal of egg yolk deposit rinsed with 5% w/v LAS solution at 80°C and 0.5 ms^{-1}

6.1.3 Deposit resistance profile, R_d

R_d was plotted when complete deposit removal was achieved. R_d was determined from the heat transfer coefficient at time (U_t) and the heat transfer coefficient when clean surface is achieved (U_c) using the following equation; (Goode, 2012)

$$R_d = \frac{1}{U_t} - \frac{1}{U_c} \quad (1)$$

Figure 6.23 shows Rd profile for cleaning of egg yolk deposit rinsed with 1 % w/v sodium hydroxide at 60^{o}C and 0.5 ms^{-1}. Lag phase starts after 500 s of experiment. The actual deposit resistance reduction starts after 5000 s and becomes zero after 10000 s. The U values depend on T_{c2}, MHFS voltage output, and the average of T_{c4} and T_{c5} (T_{av}). In this experiment, T_{av} was found to increase more rapidly than T_{c2} and MHFS voltage value which gives lower U values during the first 500 s of experiment (figure 6.3), thus it results in higher Rd values during the first 500 s. For cleaning using 1 % w/v sodium hydroxide at 80^{o}C and 0.5 ms^{-1}, result indicates that deposit resistance starts to decline rapidly after 550 s of experiment and falls to zero after 2000 s of rinsing (figure 6.24). Cleaning with 5 % w/v sodium hydroxide at 60^{o}C and 0.5 ms^{-1} is presented in figure 6.25. Lag phase starts after 400 s and the actual deposit resistance reduction was shown from 3000 s and becomes zero after 5000 s of expriment. Higher in deposit resistance during the first 400 s of experiment is caused by higher increment in T_{av} values compares to MHFS voltage output and T_{c2} values which gives lower U values (figure 6.6). Figure 6.26 illustrates R_d profile for cleaning of egg yolk deposit rinsed with 5 % w/v sodium hydroxide at 80^{o}C and 0.5 ms^{-1}. Deposit resistance starts to decline after 500 s of experiment and reaches zero after 1300 s of experiment. Figure 6.27 shows R_d profile for cleaning of egg yolk deposit by 1 % w/v LAS solution at 60^{o}C and

0.5 ms^{-1}. Lag phase is presented by 150 s to 8000 s and the actual deposit reduction starts after 8000 s and becomes zero after 10000 s of rinsing. This is caused by higher increment in T_{av} compares to MHFS voltage output and T_{c2} values during the first 150 s of experiment which gives lower U values, thus results in higher R_d values. The similar case was shown by figure 6.28 and 6.30 where lag phase is presented by 300 to 1600 s and 200 to 1600 s respectively, while actual deposit resistance reduction happens after 1600 and 1300 s respectively and falls to zero after 2800 s and 1900 s respectively. Figure 6.29 illustrates cleaning of egg yolk deposit rinsed with 5 % w/v LAS solution at 60^{o}C and 0.5 ms^{-1}. Lag phase occurs during the first 3000 s. Deposit resistance starts to decline rapidly after 3300 s of experiment and becomes zero after 5000 s of chemical rinsing.

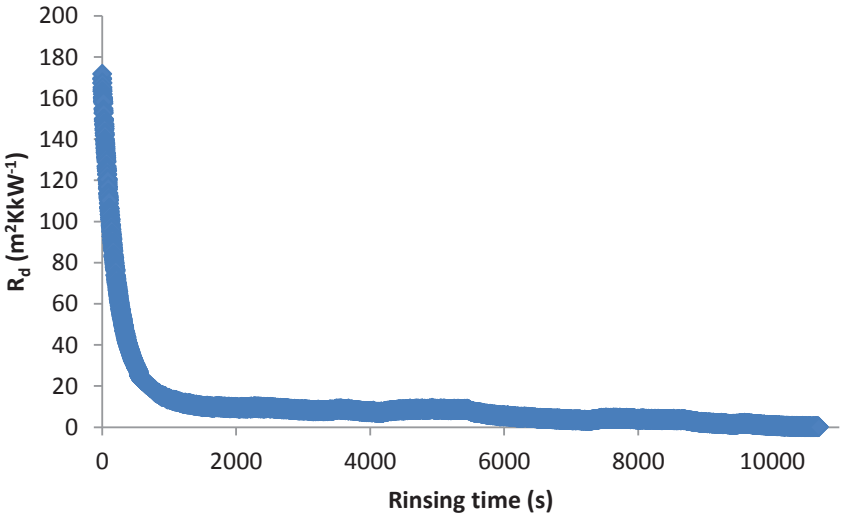


Figure 6.23: R_d profile for egg yolk deposit cleaning using 1 % w/v sodium hydroxide at 60^{o}C and 0.5 ms^{-1}

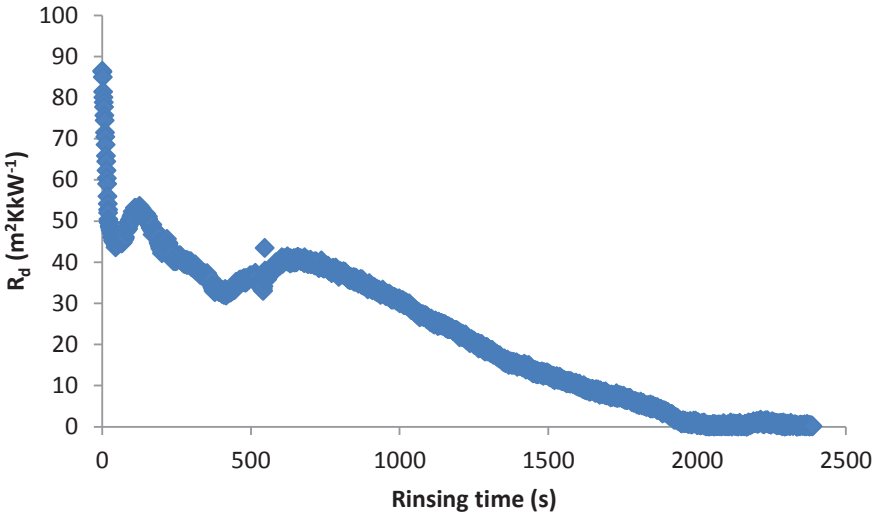


Figure 6.24: R_d profile for egg yolk deposit cleaning using 1 % w/v sodium hydroxide at 80°C and 0.5 ms^{-1}

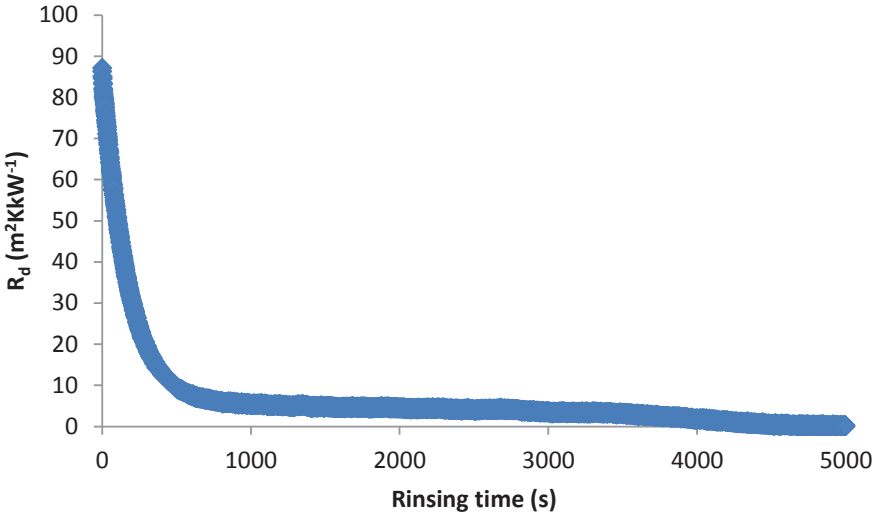


Figure 6.25: R_d profile for egg yolk deposit cleaning using 5 % w/v sodium hydroxide at 60°C and 0.5 ms^{-1}

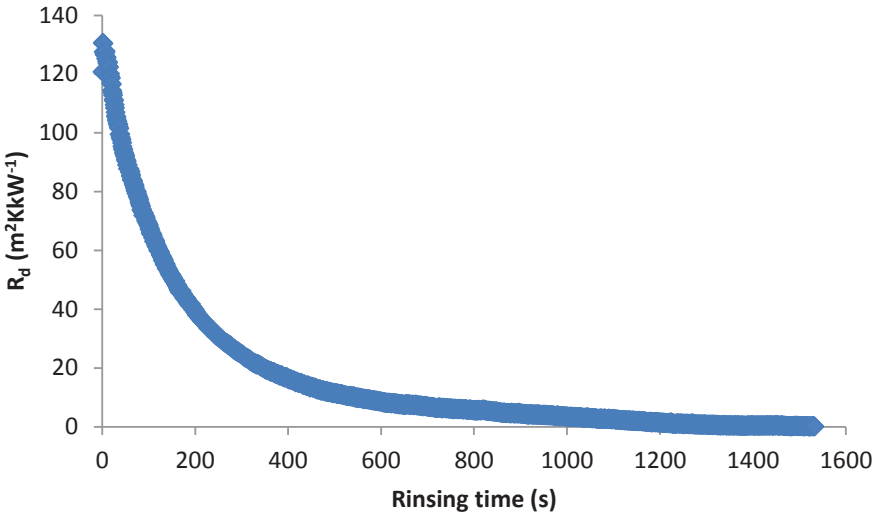


Figure 6.26: R_d profile for egg yolk deposit cleaning using 5 % w/v sodium hydroxide at 80°C and 0.5 ms^{-1}

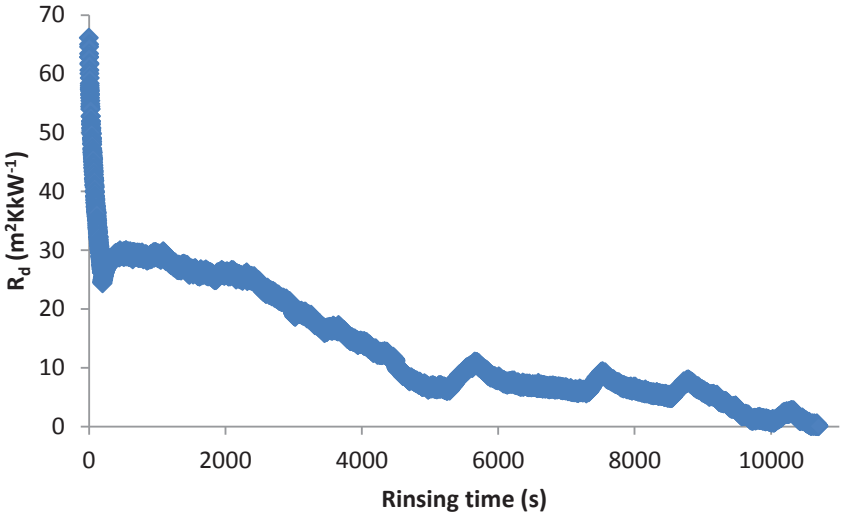


Figure 6.27: R_d profile for egg yolk deposit cleaning using 1 % w/v LAS solution at 60°C and 0.5 ms^{-1}

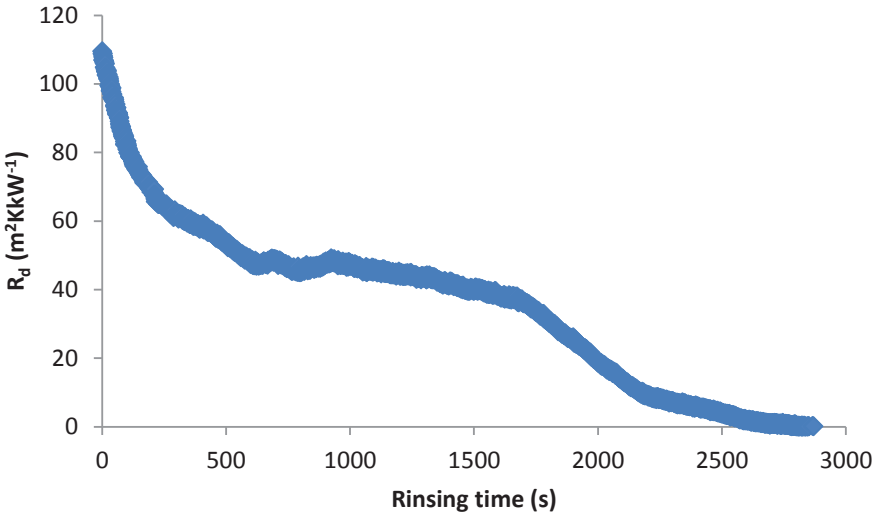


Figure 6.28: R_d profile for egg yolk deposit cleaning using 1 % w/v LAS solution at 80°C and 0.5 ms^{-1}

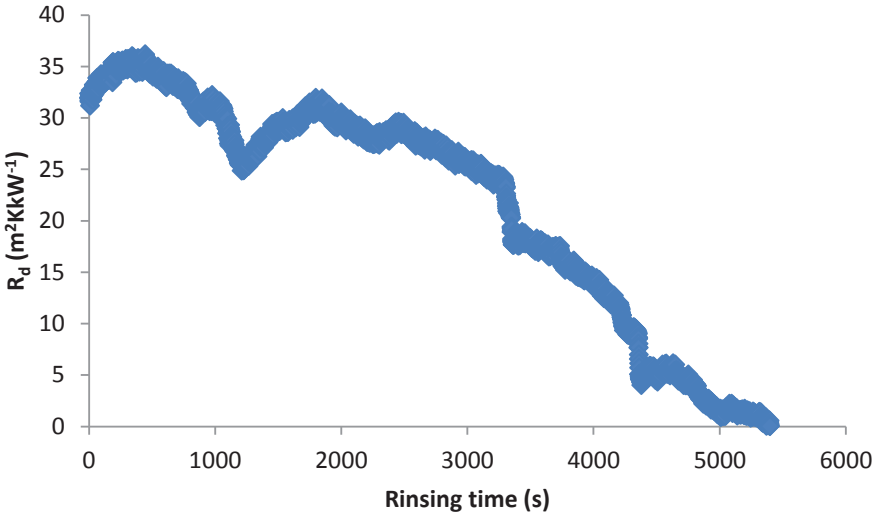


Figure 6.29: R_d profile for egg yolk deposit cleaning using 5 % w/v LAS solution at 60°C and 0.5 ms^{-1}

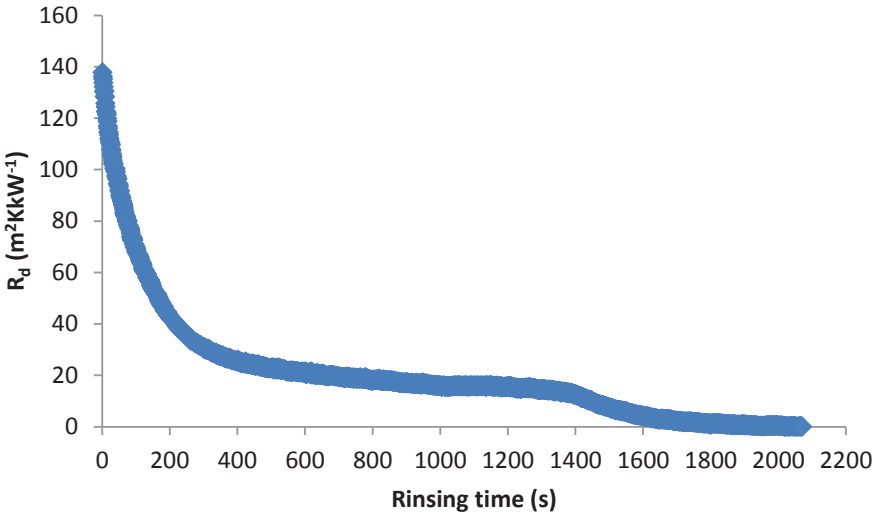


Figure 6.30: R_d profile for egg yolk deposit cleaning using 5 % w/v LAS solution at 80°C and 0.5 ms^{-1}

6.2 Discussions

6.2.1 Effect of temperature on cleaning of egg yolk deposits

For egg yolk samples rinsed with 1 % w/v sodium hydroxide, increase in temperature from 40°C to 60°C decreased the lag phase duration by 16.67 %. Increasing the temperature from 60°C to 80°C shortened the lag phase duration by 90 %, reduced the removal phase by 65 % and decreased the visual cleaning time by 81.14 %. The lag phase for cleaning of egg yolk with 5 % w/v sodium hydroxide was found to be shortened by 63.89 % when temperature was increased from 40°C to 60°C. Increasing the temperature from 60°C to 80°C decrease the lag phase length by 76.92 %, reduced the removal phase duration by 73.68 % and shortened the visual cleaning time by 70.67 %. For egg yolk samples rinsed with 1 % w/v LAS solution, increase in temperature from 40°C to 60°C surprisingly increased the lag phase duration by 11.11 %. When temperature was increased from 60°C to 80°C, the lag phase was found to decrease by

80 %, the removal phase was reduced by 47.83 % and 72.67 % of the cleaning time was reduced. Cleaning of egg yolk deposit with 5 % LAS solution shows 54.17 % decrease in lag phase duration when the temperature was increase from 40^{o}C to 60^{o}C. Increasing the temperature from 60^{o}C to 80^{o}C shortened the lag phase length by 60.61 %, decreased the removal phase duration by 70 % and reduced the cleaning time 73.33 %. Thus, it can be concluded that increase the temperature helps to reduce the lag phase duration, the removal phase duration and the cleaning time except for cleaning of egg yolk deposit with 1 % w/v LAS solution where increasing the temperature from 40^{o}C to 6^{o}C increased the lag phase by 11.11 %.

Christian (2004) found a linear relationship between increase in temperature and decrease in cleaning time of tomato paste deposit. Cleaning time was minimized by increasing the temperature from 30 to 70^{o}C. Increasing the temperature from the lowest temperature to the highest temperature shortened the cleaning time by 6 times. For the cleaning of WPC deposit, researcher found no deposit removal at all temperatures (30 – 70^{o}C) used without chemical action. At 0.5% caustic solution, increase in temperature gave shorter cleaning time. Aziz (2008) reported no albumin deposit removal without chemical cleaning. Temperature plays a major role in the cleaning of egg albumin deposit as it can affect the strength of chemical concentration and flow velocity effect. Increase in temperature decreased the cleaning time. Othman et al (2010) stated that temperature affected the cleaning behavior of sweet condensed milk. At all temperatures tested, the cleaning was reduced by increasing the flow rate from 0.25 to 0.5 m s^{-1}. A linear relationship was found between increase in temperature from 40 to 80^{o}C and decrease in cleaning time. Studies by Cole (2011) and Palabiyik (2013) also

reported that increase in temperature at all flow rates reduced the cleaning time of toothpaste. Goode (2012) found that the effect of different temperatures on cleaning appeared to be less significant on cleaning time of yeast slurry deposit. However, increase in temperature reduced the lag phase and removal phase duration of yeast deposit slurry cleaning. The effect of temperature was found to be greater than the effect of chemical concentration on the cleaning of aged yeast slurry deposit. At 0.2 % chemical concentration, increasing the temperature from 30 to 70^{o}C at 0.26 ms^{-1} shortened the removal phase duration. For the cleaning of cooked caramel deposit, it was found that complete removal was only achieved at 80^{o}C. Rois Anwar (2016) suggests that no complete deposit removal was seen without chemical action even at the highest temperature (70^{o}C) and flow rate (0.5 ms^{-1}). This agrees with the present study result that increasing the temperature helps to reduce the cleaning time of egg yolk deposit.

6.2.2 Effect of chemical concentration on cleaning of egg yolk deposit

For cleaning of egg yolk deposit rinsed sodium hydroxide and LAS solution at 40^{o}C, the lag phase duration was not affected by increasing the concentration from 1 to 5 %. Increasing the sodium hydroxide concentration from 1 to 5% at 60^{o}C decreased the lag phase length of egg yolk deposit cleaning by 56.67 %, reduced the removal phase duration by 52.5 % and decreased the cleaning time by 57.14 %. For egg yolk samples rinsed with LAS solution at 60^{o}C, the lag phase duration was reduced by 58.75 % when the concentration was increased from 1 to 5 %, while the removal phase length and the cleaning time was decreased by 15 and 32.23 % respectively when the concentration

was raised from 1 to 5 %. Increasing the concentration of sodium hydroxide from 1 to 5 % at 80^{o}C did not affect the lag phase duration of egg yolk deposit cleaning, but decreased the removal phase by 64.29 % and reduced the cleaning time by 33.33 %. At 80^{o}C, increase in concentration of LAS solution from 1 to 5 % reduced the lag phase duration by 18.75 %, decreased the removal phase length by 50 % and reduced the visual cleaning time of egg yolk deposit by 31.91 %. Therefore it can be concluded that increasing the concentration from 1 to 5 % helps to decrease the lag phase duration, the removal phase length and the visual cleaning time of egg yolk deposit except for the cleaning of egg yolk deposit rinsed with sodium hydroxide and LAS solution at 40^{o}C and cleaning of egg yolk deposit rinsed with sodium hydroxide at 80^{o}C where increase in concentration from 1 to 5 % did not affect the lag phase duration.

Christian (2004) reported no deposit removal at all temperatures (30 – 70^{o}C) and flow rates (0.7 to 2.3 l min^{-1}) used without chemical action for the cleaning of WPC deposit and concluded that the effect of temperature is greater than flow rate and chemical concentration in the cleaning of WPC deposit. Aziz (2008) stated that significant decrease in cleaning time was normally shown at initial increase in chemical concentration and no optimum chemical concentration was found at 50^{o}C and 1.5 l min^{-1} flow velocity. Generally, increase in chemical concentration decreased the cleaning time. Othman *et al.* (2010) found no significant effect by increasing the caustic solution concentration from 0.5 to 1.5 %. Goode (2012) stated that complete removal of aged yeast slurry was achieved by chemical cleaning with the effect of temperature and chemical concentration was clearly seen at low flow rate while at higher flow rate, the effect of temperature was greater than the effect of chemical concentration. Rois Anwar

(2016) reports No complete deposit removal was seen without chemical action even at the highest temperature (70^{o}C) and flow rate (0.5 ms^{-1}). At 0.1% caustic solution, complete removal of deposit fouled on polycarbonate was found at 40, 55 and 70^{o}C. This is aligned with the current findings that chemical concentration plays an important role in the cleaning of egg yolk deposit.

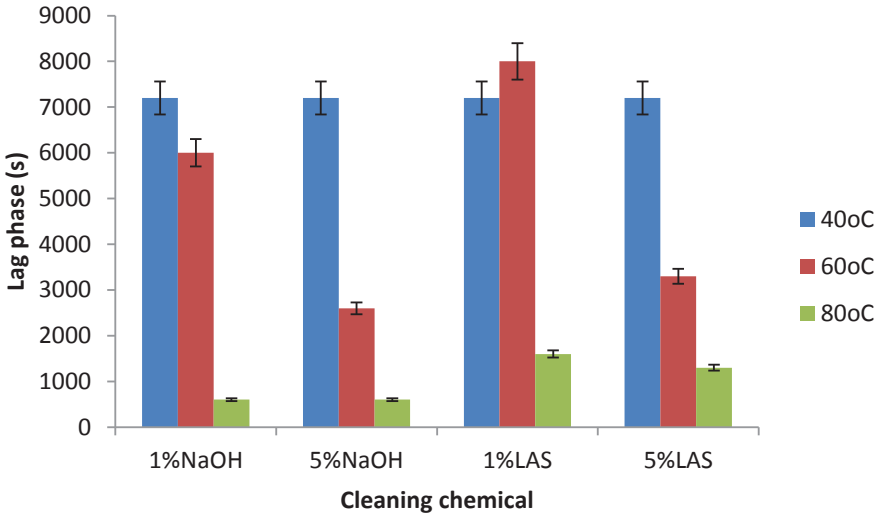


Figure 6.31: Duration of the lag phase in the cleaning of egg yolk deposit

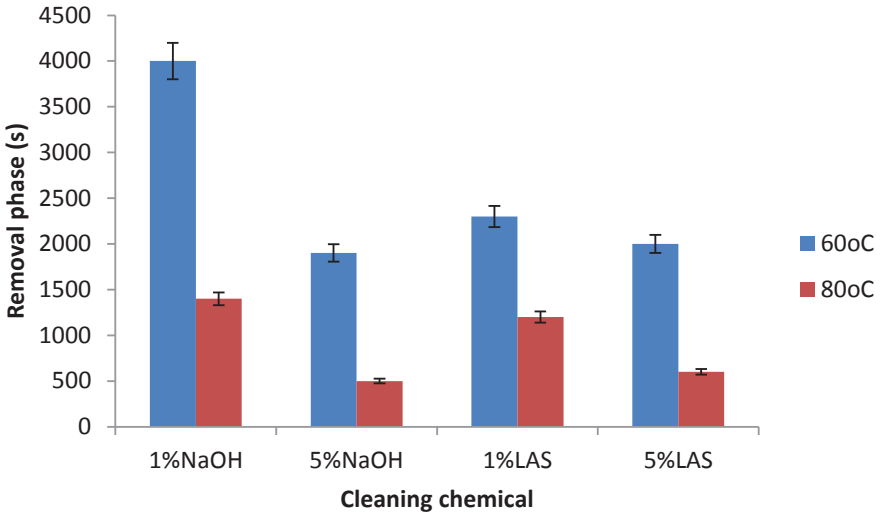


Figure 6.32: Duration of the removal phase in the cleaning of egg yolk deposit

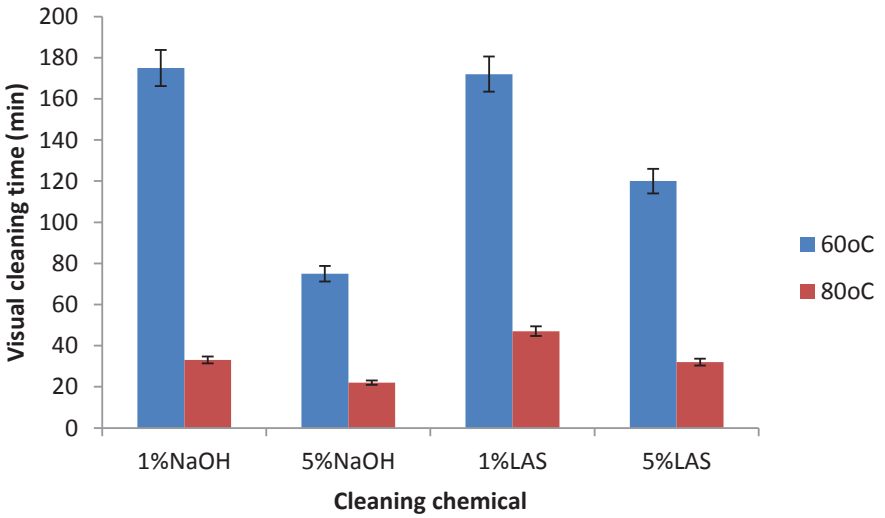


Figure 6.33: Visual cleaning time of egg yolk deposit

6.23 Cleaning of egg yolk deposits by image analysis

Microscope camera was used in order to capture images for determination of deposit removal by image analysis. Figure 6.34 shows the images for the cleaning of egg yolk deposit rinsed with 1% w/v sodium hydroxide at 60^{o}C and 0.5 ms^{-1}. This cleaning can be divided into four stages;

Stage 1 (0 to 135 min) - Cleaning of egg yolk deposit by cohesive failure occurred where the top layer of the deposit is gradually removed which results in the change in colour of deposit from brownish yellow to light yellow colour.

Stage 2 (135 to 155 min) - Removal of egg yolk deposit at the coupon edge caused by edge effect. The deposit thickness at the coupon edge becomes thinner until it can be removed by physical action of cleaning fluid.

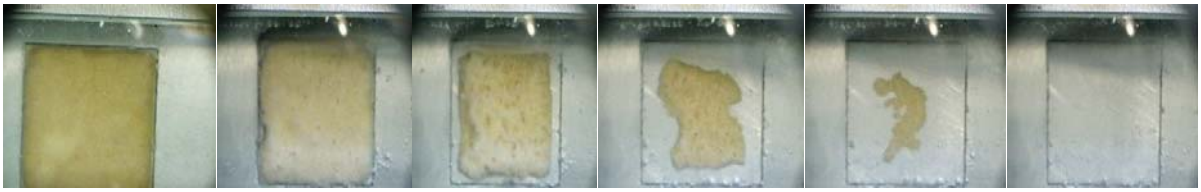
Stage 3 (155 to 170 min) – Extension of deposit erosion from the fracture point at the coupon edge together with the removal of the top layer of the deposit due to cohesive failure.

Stage 4 (170 min) – Egg yolk deposit is totally removed from the stainless steel surface.

This similar behaviour was also shown in figure 6.35 for the cleaning of egg yolk deposit rinsed with 5 % LAS solution at 80^{o}C and 0.5 ms^{-1}, where 0 to 15 minute of experiment represents stage 1, 15 to 35 minute of rinsing represents stage 2, 35 to 50 minute of experiment represents stage 3 and stage 4 was achieved at 50 minutes of rinsing.

0 min 135 min 145 min 155 min 165 min 170 min

Figure 6.34: Cleaning of egg yolk deposit rinsed with 1 % w/v sodium hydroxide at 60^{o}C and 0.5 ms^{-1}



0 min 15 min 25 min 35 min 40 min 50 min

Figure 6.35: Cleaning of egg yolk deposit rinsed with 1 % w/v LAS solution at 80^{o}C and 0.5 ms^{-1}

6.2.4 Heat transfer coefficient (U) profile vs. Image analysis

Both techniques were used in this research to study the cleaning progress of egg yolk deposit on stainless steel coupon. Both experiments are carried out simultaneously in order to compare the results from both techniques used. In most cases, the cleaning time from both sets of experiment was seems to be comparable. However, there is a limitation shown by the image analysis technique as it is hard to determine the deposit layer removal at the early stage of experiment. Results from the U profile were seen to be more accurate compares to results from image analysis.

6.2.5 Typical behaviour of scrapping process

Micromanipulation technique was used in this study in order to determine the force required to remove the egg yolk deposit from the stainless steel surface. The force was measured when the micromanipulator arm was drawn horizontally to move the T-shaped probe across the deposit and scrap the deposit off the surface. Figure 6.36 shows the typical curve for force measured vs sampling time. Initially, the T-shaped probe moves towards the deposit at point A, where the T-shaped probe starts to contact the deposit and the scrapping process begins. The force measured increases until it reaches its maximum point at point B then the force measured starts to fall gradually

until it reaches zero at point C. At point C, T-shaped probe was observed to be at the edge of the surface and moves away from the surface (point D) where no force was recorded from point C to point D as the probe pulls the deposit away from the surface. For adhesive strength measurement, the gap between the probe and the surface is set to be at the lowest gap (10 μm) so that no deposit layer left on the surface after experiment, while for cohesive strength measure, the gap between the probe and the deposit is set to be higher leaving a deposit layer on the surface after scrapping process done.

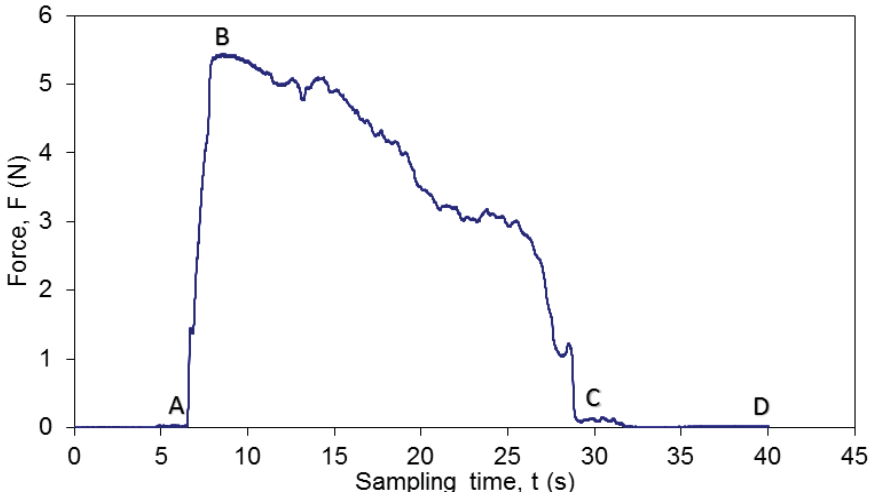


Figure 6.36: Typical curve of force versus sampling time for the pulling of egg yolk deposit by T-shaped probe.

6.2.6 Determination of adhesive strength of egg yolk deposits

In this set of experiments, egg yolk deposits were soaked at water, sodium hydroxide (1 and 5 % w/v) and LAS solution (1 and 5 % w/v) at different hydration time (15 and 30 minutes) and different temperature (20, 40, and 60^{o}C) prior to force measurement. For samples soaked in water at 15 minutes hydration time (figure 6.37), deposits soaked at 60^{o}C gives the lowest adhesive strength (189.71 $\pm$ 2.02 Jm^{-2}) followed by deposits soaked at 40^{o}C (195.46 + 2.17 Jm^{-2}) and deposits soaked at 20^{o}C have the highest adhesion strength (196 $\pm$ 1.85 Jm^{-2}). No significant difference was shown between the experiments with different hydration temperature (p=0.104, 0.109). Figure 6.38 shows the adhesive strength of egg yolk deposits soaked in 1 % w/v sodium hydroxide solution at 15 minutes hydration time. Samples hydrated at 20^{o}C (160.5 $\pm$ 1.49 Jm^{-2}) have the highest adhesive force followed by samples soaked at 40^{o}C (150.18 $\pm$ 1.72 Jm^{-2}) and samples soaked at 60^{o}C (138.26 $\pm$ 1.53 Jm^{-2}). Figure 6.39 illustrates the adhesive strength of egg yolk deposit soaked in 1 % LAS solution at 15 minutes hydration time. It was found that samples hydrated at 20^{o}C give the highest adhesive strength (161.42 $\pm$ 2.36 Jm^{-2}) followed by samples hydrated at 40^{o}C (1.55.43 $\pm$ 2.09 Jm^{-2}) and the lowest adhesive strength was given by samples soaked at 60^{o}C (141.82 $\pm$ 1.86 Jm^{-2}). There was no significant difference between the experiments at 20^{o}C and 40^{o}C hydration temperature (p=0.0752). The adhesive strength of egg yolk deposit soaked in 5 % sodium hydroxide at 15 minutes hydration time was shown in figure 6.40. Samples soaked at the lowest temperature (20^{o}C) have the highest adhesive strength (142.03 $\pm$ 1.79 Jm^{-2}), while samples soaked at the highest temperature (60^{o}C) gives the lowest adhesive strength (122.3 $\pm$ 1.55 Jm^{-2}). Samples soaked at 40^{o}C show adhesive strength

(127.14 ± 138 Jm^{-2}). However, no significant different was shown between the experiments at 40°C and 60°C hydration temperature (p=0.0602). Figure 6.41 illustrates the adhesive strength of egg yolk deposit hydrated in 5 % LAS solution at 15 minutes hydration time. The similar trend continues where the highest hydration temperature (60°C) results in the lowest adhesive strength (130.81 ± 2.57 Jm^{-2}) followed by samples soaked at 40°C (149.96 ± 2.25 Jm^{-2}) and samples soaked at 20°C which gives the highest adhesive strength (154.83 ± 2.74 Jm^{-2}). There was no significant difference between the experiments at 20°C and 40°C hydration temperature (p=0.084).

Figure 6.42 shows the adhesive strength of egg yolk deposit hydrated in water at 30 minutes hydration time. The highest adhesive was shown by samples soaked at 20°C (190.36 ± 2.86 Jm^{-2}) followed by samples soaked at 40°C (163.81 ± 1.82 Jm^{-2}) and samples hydrated at 60°C (150.1 ± 1.63 Jm^{-2}). The adhesive strength of egg yolk deposit soaked in 1 % w/v sodium hydroxide at 30 minutes hydration time was shown in figure 6.43. The similar trends is found where samples soaked at the highest temperature (60°C) have the lowest adhesive strength (113.52 ± 1.28 Jm^{-2}) and samples soaked at the lowest temperature (20°C) gives the highest adhesive strength (131.63 ± 149 Jm^{-2}). Samples hydrated at 40°c shows 122.11 ± 1.51 Jm^{-2} adhesive strength. Figure 6.44 illustrates the egg yolk deposit adhesive strength hydrated in 1 % LAS solution at 30 minutes hydration time. It happened that samples soaked at 60°C have the lowest adhesive strength (115.53 ± 1.93 Jm^{-2}) compares to samples soaked at 40°C (147.68 ± 2.26 Jm^{-2}) and samples hydrated at 20°C (152.65 ± 2.11 Jm^{-2}). No significant difference was shown between the experiments at 20°C hydration and the experiments at 40°C hydration temperature (p=0.083). Figure 6.45 and 6.46 follows the

similar trend where the lowest temperature give the highest adhesive strength for samples soaked in 5 % w/v sodium hydroxide and LAS solution at 30 minutes hydration time (121.73 ± 1.43 and 143.05 ± 1.67 Jm^{-2}), while samples soaked at 40°C have 116.55 ± 1.38 and 142.13 ± 1.82 Jm^{-2} adhesion strength and samples soaked at 60°C show 104.24 ± 1.53 and 113.95 ± 1.47 Jm^{-2} adhesion strength. There was no significant difference between the experiments at 20°C and 40°C sodium hydroxide pre-soaking temperature (p=0.055, 0.926).

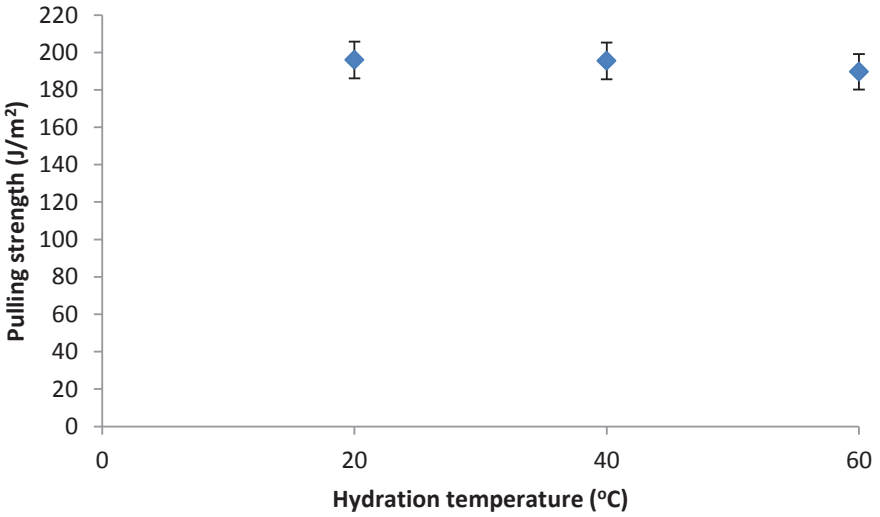


Figure 6.37: Adhesive strength of egg yolk deposit soaked in water at 15 minutes hydration time

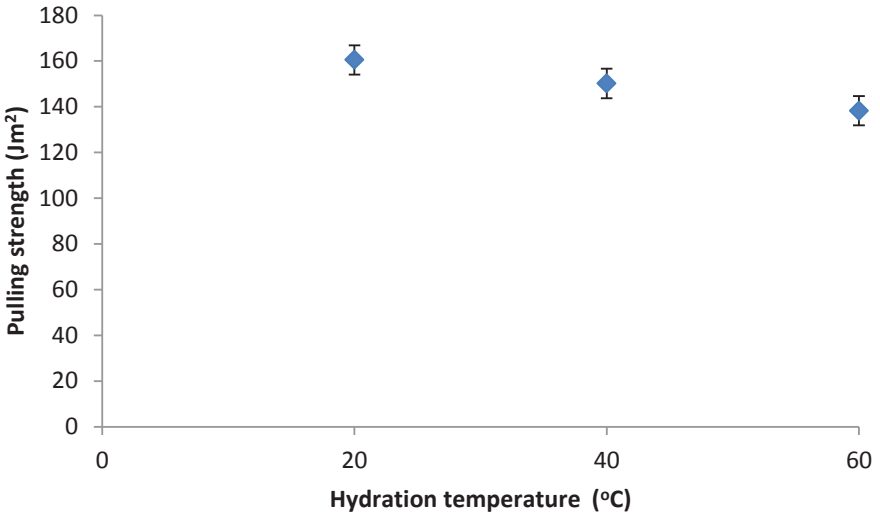


Figure 6.38: Adhesive strength of egg yolk deposit soaked in 1 % w/v sodium hydroxide at 15 minutes hydration time

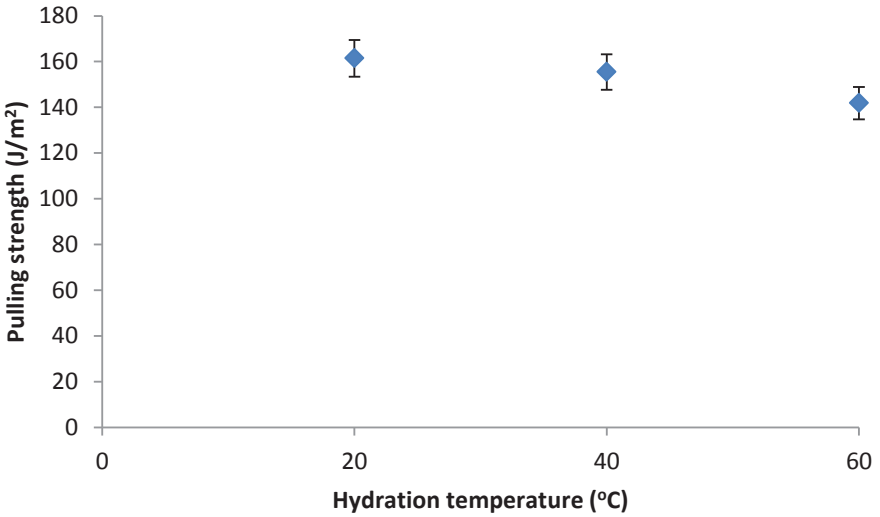


Figure 6.39: Adhesive strength of egg yolk deposit soaked in 1 % w/v LAS solution at 15 minutes hydration time

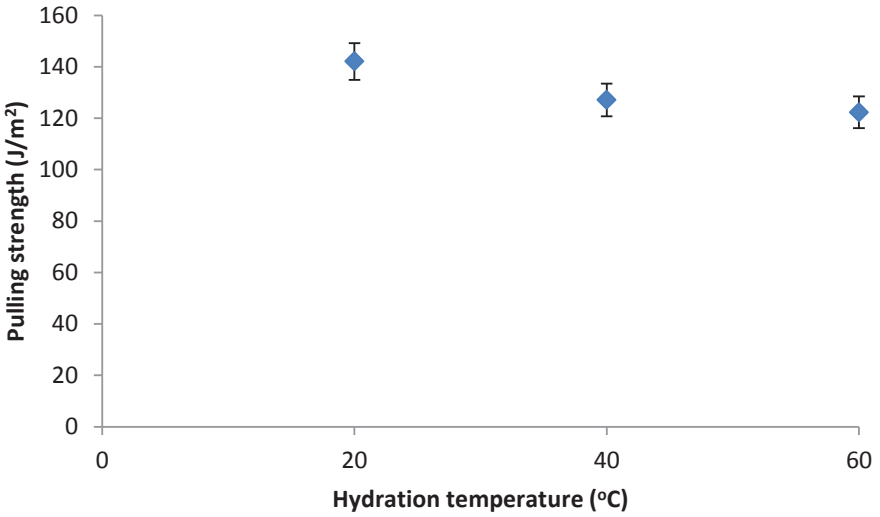


Figure 6.40: Adhesive strength of egg yolk deposit soaked in 5 % w/v sodium hydroxide at 15 minutes hydration time

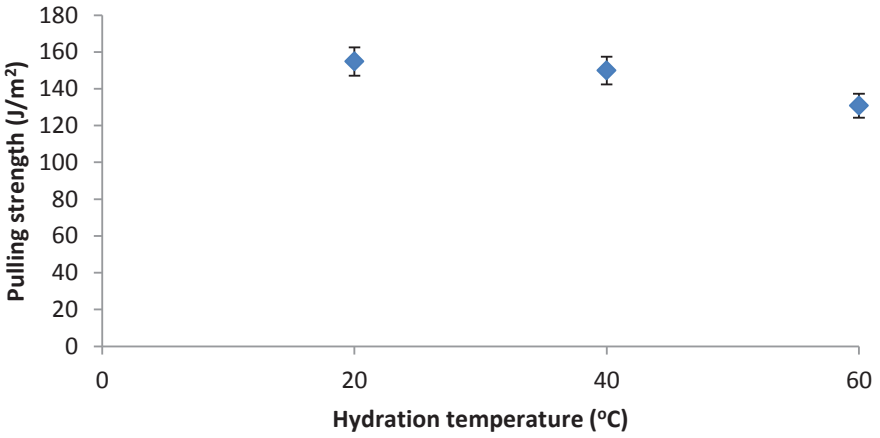


Figure 6.41: Adhesive strength of egg yolk deposit soaked in 5 % w/v LAS solution at 15 minutes hydration time

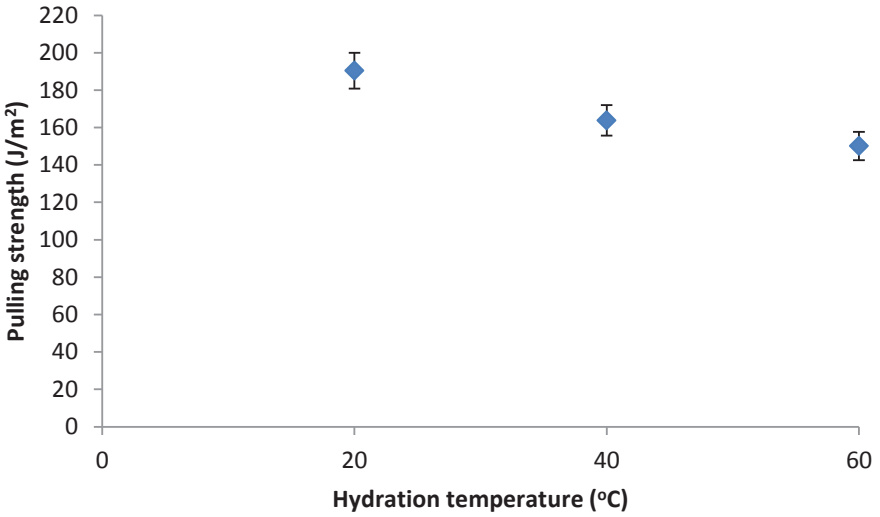


Figure 6.42: Adhesive strength of egg yolk deposit soaked in water at 30 minutes hydration time

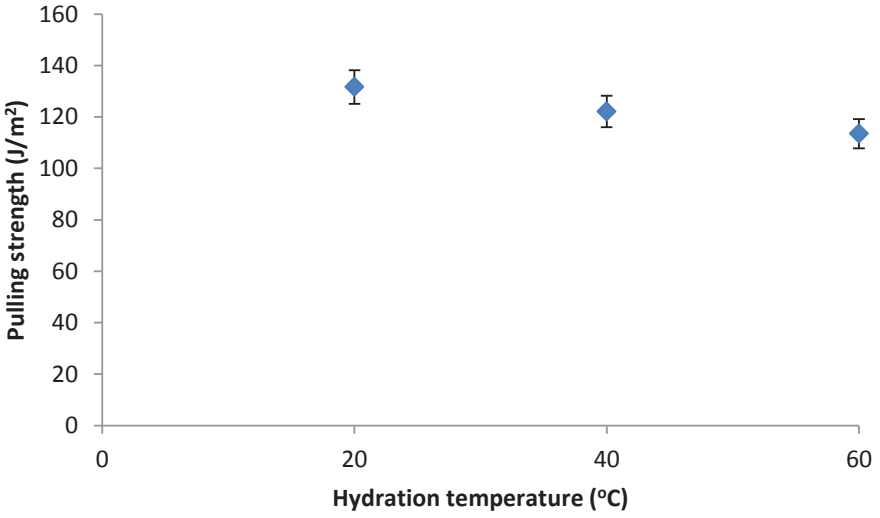


Figure 6.43: Adhesive strength of egg yolk deposit soaked in 1 % w/v sodium hydroxide at 30 minutes hydration time

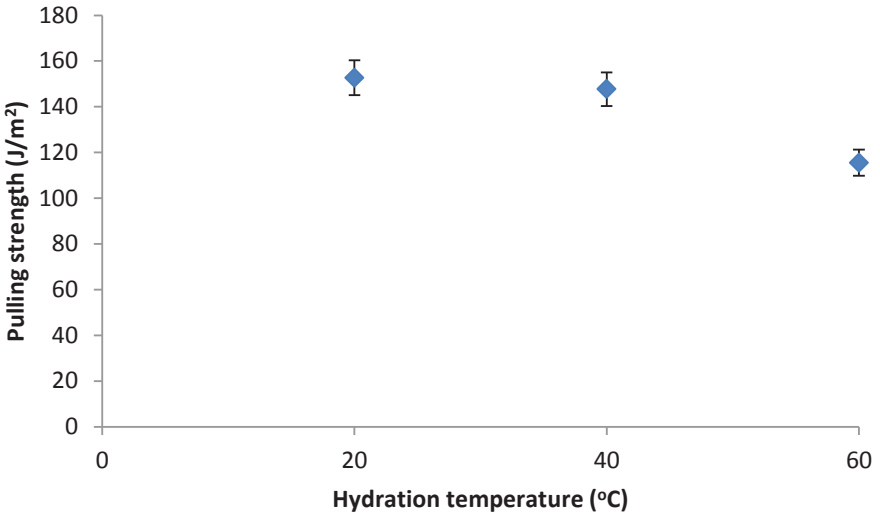


Figure 6.44: Adhesive strength of egg yolk deposit soaked in 1 % w/v LAS solution at 30 minutes hydration time

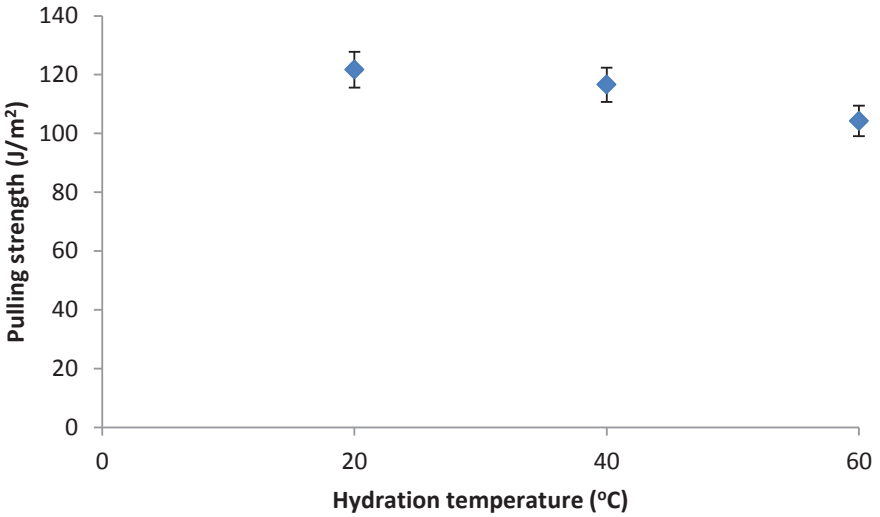


Figure 6.45: Adhesive strength of egg yolk deposit soaked in 5 % w/v sodium hydroxide at 30 minutes hydration time

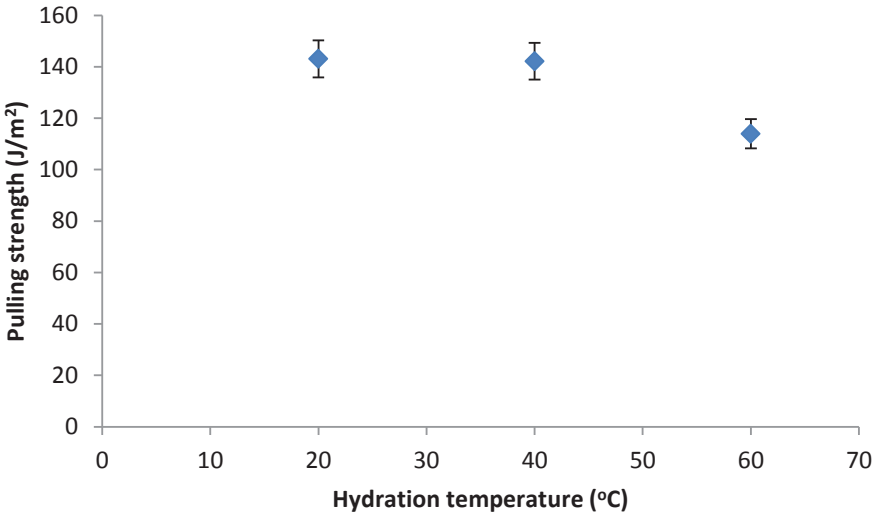


Figure 6.46: Adhesive strength of egg yolk deposit soaked in 5 % w/v LAS solution at 30 minutes hydration time

6.2.7 Determination of cohesive strength of egg yolk deposit

The gap between the T-shaped probe and the surface was raised to 1 mm in order to measure the cohesive strength of deposits. The top layer of deposits was scrapped off the surface leaving a deposit layer on the surface. The deposits were pre-soaked in different water, sodium hydroxide and LAS solution at different temperatures, concentrations and hydration time prior to measurement. Figure 6.47 illustrates the cohesive strength of the egg yolk deposit soaked in water at 15 minutes hydration time. Samples soaked in water at 20^{o}C show the highest cohesive strength (119.83 $\pm$ 2.03 Jm^{-2}) followed by samples hydrated at 40^{o}C (113.21 $\pm$ 1.57 Jm^{-2}) and samples soaked at 60^{o}C (108.18 $\pm$ 1.39 Jm^{-2}). No significant difference was shown between the experiments with different hydration temperature (p=0.063, 0.076). The cohesive strength of egg yolk deposits hydrated in 1 % w/v sodium hydroxide was shown in figure

6.48. As expected, samples soaked at 60^{o}C in 1 % w/v sodium hydroxide give the lowest cohesive strength (90.19 $\pm$ 1.26 Jm^{-2}). Samples soaked at 40^{o}C show (99.67 $\pm$ 1.19 Jm^{-2}) cohesive strength and samples soaked at 20^{o}C have 108.17 $\pm$ 1.42 Jm^{-2} cohesive strength. There was no significant difference between the experiments at 20^{o}C and 40^{o}C hydration temperature (p=0.079). Figure 6.49 represents the cohesive strength of egg yolk deposits soaked in 1 % LAS solution at 15 minutes hydration time. Samples soaked at 20^{o}C give 117.08 $\pm$ 1.24 Jm^{-2} cohesive strength while samples soaked at 40^{o}C have 107.44 $\pm$ 1.73 Jm^{-2} and samples soaked 60^{o}C were found to have the lowest cohesive strength (102.59 $\pm$ 1.81 Jm^{-2}). No significant difference was shown between the experiments at 40^{o}C and 60^{o}C hydration temperature (p=0.099). The similar trend was observed in the cohesive strength of egg yolk deposits hydrated in 5 % w/v sodium hydroxide and LAS solution at 15 minutes hydration (figure 6.50 and 6.51) where deposits soaked at the highest temperature (60^{o}C) have the lowest cohesive strength (81.96 $\pm$ 1.17 and 96.18 $\pm$ 1.31 Jm^{-2}) and deposits hydrated at the lowest temperature (20^{o}C) give the highest cohesive strength (101.44 $\pm$ 1.78 and 113.04 $\pm$ 2.01 Jm^{-2}). Samples soaked at 40^{o}C give 94.06 $\pm$ 1.57 and 101.32 $\pm$ 1.39 Jm^{-2} cohesive strength. There was no significant difference between the experiments at 40^{o}C and 60^{o}C LAS solution pre-soaking temperature (p=0.085) in figure 6.51.

Figure 6.52 illustrates the cohesive strength of egg yolk deposits soaked in water at 30 minutes hydration time. It was found that samples soaked in water at 20^{o}C have the highest cohesive strength (114.85 $\pm$ 1.69 Jm^{-2}) followed by deposits hydrated at 40^{o}C (102.02 $\pm$ 1.48 Jm^{-2}) and samples soaked at 60^{o}C (100.18 $\pm$ 1.34 Jm^{-2}). No significant difference was shown between the experiments at 40^{o}C and 60^{o}C hydration

temperature (p=0.472). The cohesive strength of samples soaked in 1 % w/v sodium hydroxide at 30 minute hydration time was shown in figure 6.53. Samples soaked at the highest temperature (60^{o}C) give the lowest cohesive strength (88.21 $\pm$ 1.32 Jm^{-2}) as expected while deposits hydrated at the lowest temperature (20^{o}C) give the highest cohesive strength (103.16 $\pm$ 1.51 Jm^{-2}). There was no significant difference between the experiments at different hydration temperature (p=0.064, 0.069). The cohesive strength of samples soaked at 40^{o}C was found to be 96.36 $\pm$ 1.66 Jm^{-2}. Figure 6.54 represents the cohesive strength of egg yolk deposit hydrated in 1 % w/v LAS solution at 30 minutes hydration time. The similar trend was found in this set of experiment where samples soaked at 20^{o}C show the highest cohesive strength (108.11 $\pm$ 2.19 Jm^{-2}) followed by samples soaked at 40^{o}C (99.34 $\pm$.1.91 Jm^{-2}) and deposits hydrated at 60^{o}C (94.29 $\pm$ 1.18 Jm^{-2}). This similar trend continues in the cohesive strength of egg yolk deposits soaked in 5 % w/v sodium hydroxide and LAS solution at 30 minutes hydration time. No significant difference was shown between the experiments at different hydration temperature (p=0.053, 0.066). The highest hydration temperature (60^{o}C) appears to have the lowest deposit cohesive strength (82.07 $\pm$ 1.65 and 88.47 $\pm$ 1.49 Jm^{-2}) while, the lowest hydration temperature (20^{o}C) appears to give the highest deposit cohesive strength (95.69 $\pm$ 1.31 and 105.67 $\pm$ 1.82 Jm^{-2}). Samples soaked at 40^{o}C show 88.88 $\pm$ 2.03 and 93.45 $\pm$ 1.5 Jm^{-2} cohesive strength. There was no significant difference between the experiments at 40^{o}C and 60^{o}C LAS solution pre-soaking temperature (p=0.075) in figure 6.56.

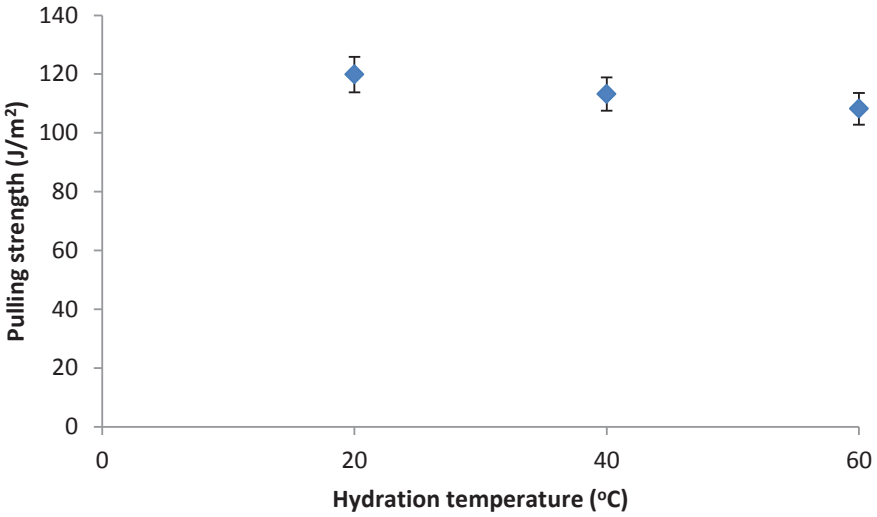


Figure 6.47: Cohesive strength of egg yolk deposit soaked in water at 15 minutes hydration time

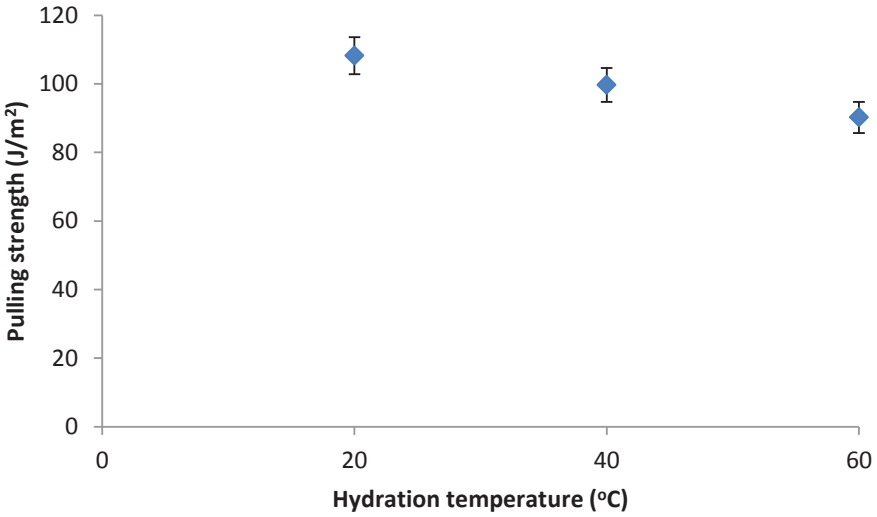


Figure 6.48: Cohesive strength of egg yolk deposit soaked in 1 % w/v sodium hydroxide at 15 minutes hydration time

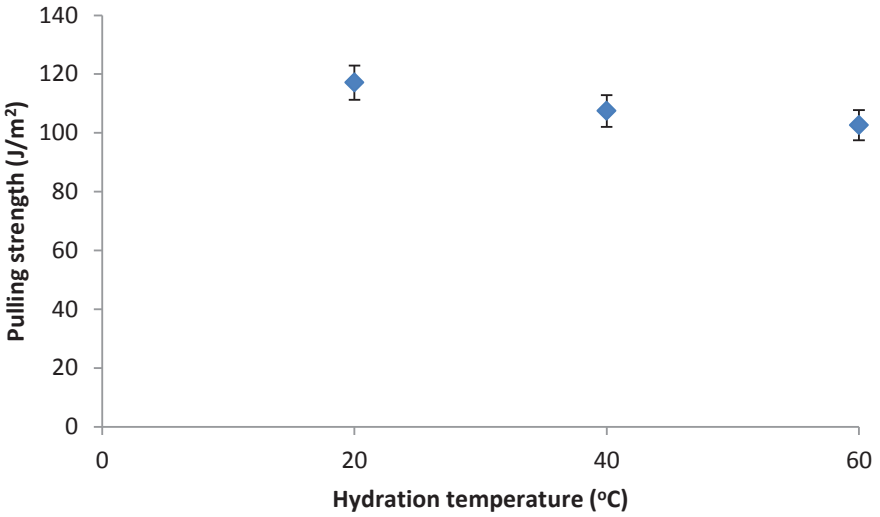


Figure 6.49: Cohesive strength of egg yolk deposit soaked in 1 % w/v LAS solution at 15 minutes hydration time

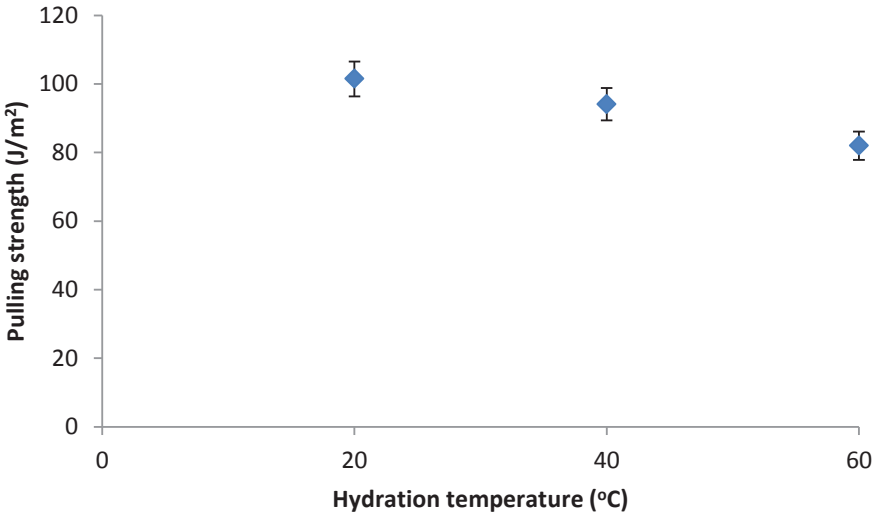


Figure 6.50: Cohesive strength of egg yolk deposit soaked in 5 % w/v sodium hydroxide at 15 minutes hydration time

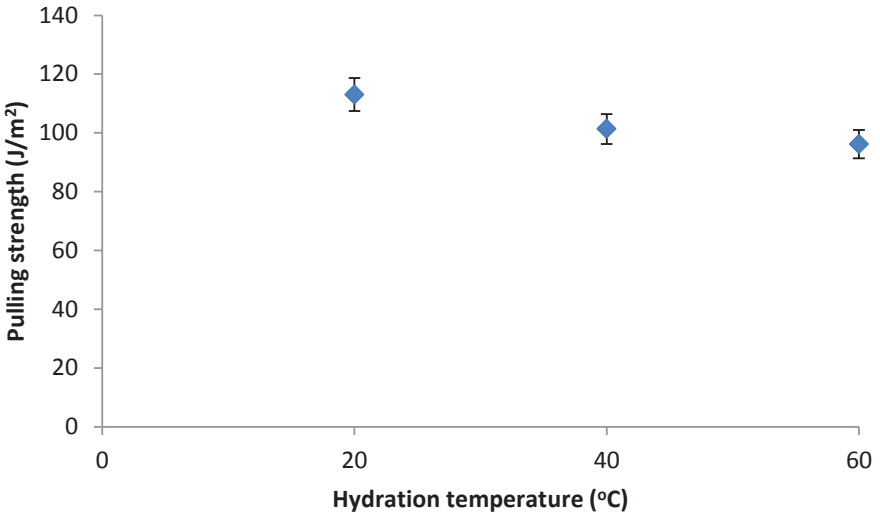


Figure 6.51: Cohesive strength of egg yolk deposit soaked in 5 % w/v LAS solution at 15 minutes hydration time

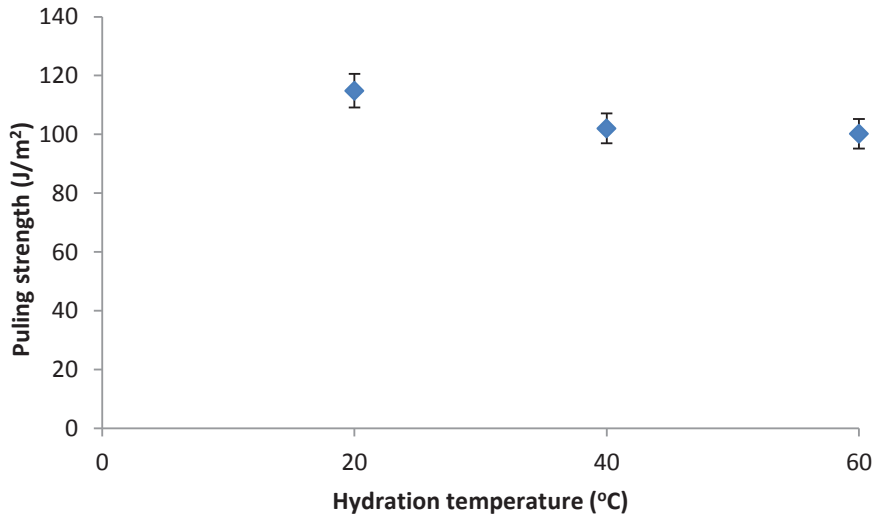


Figure 6.52: Cohesive strength of egg yolk deposit soaked in water at 30 minutes hydration time

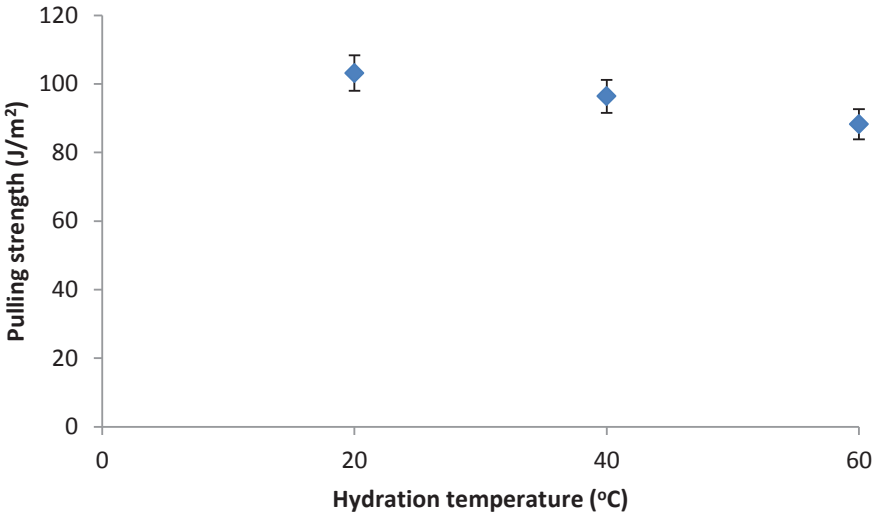


Figure 6.53: Cohesive strength of egg yolk deposit soaked in 1 % w/v sodium hydroxide at 30 minutes hydration time

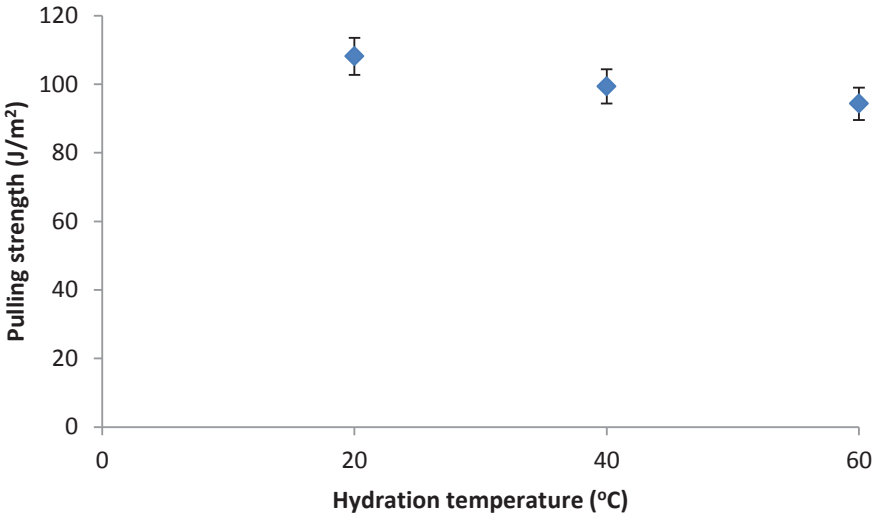


Figure 6.54: Cohesive strength of egg yolk deposit soaked in 1 % w/v LAS solution at 30 minutes hydration time

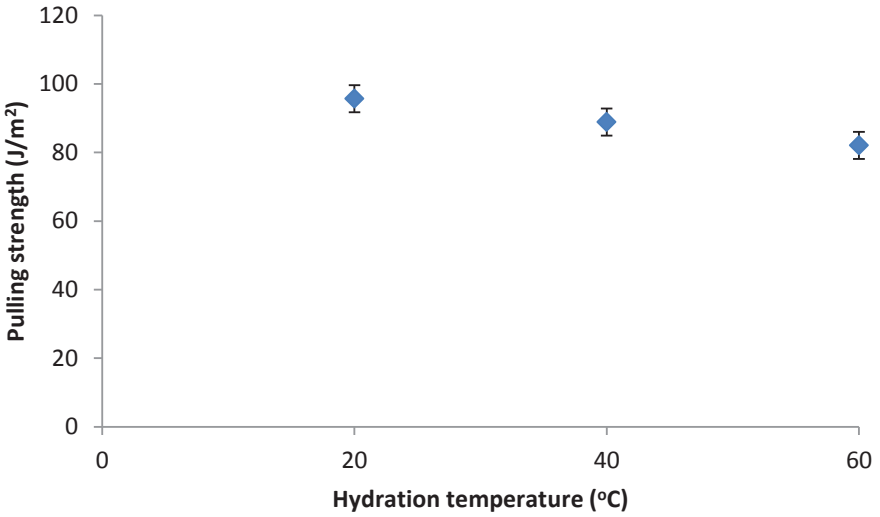


Figure 6.55: Cohesive strength of egg yolk deposit soaked in 5 % w/v sodium hydroxide at 30 minutes hydration time

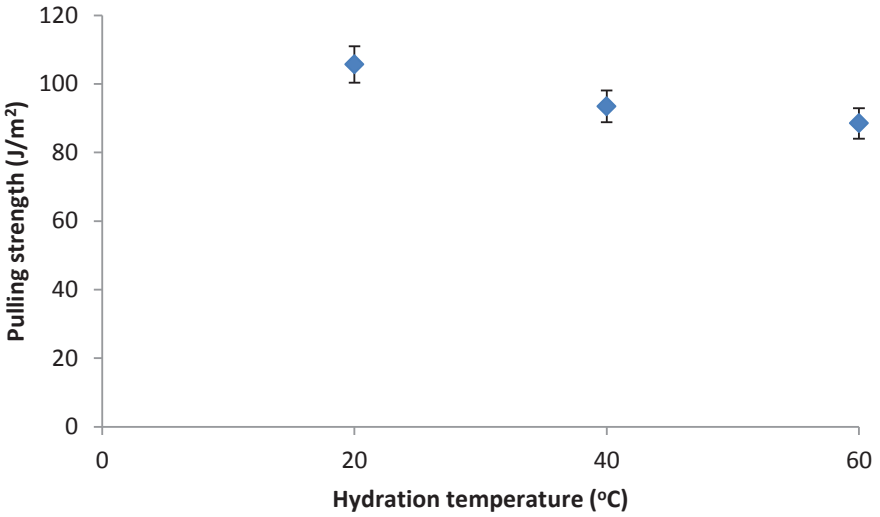


Figure 6.56: Cohesive strength of egg yolk deposit soaked in 5 % w/v LAS solution at 30 minutes hydration time

6.2.8 The effect of temperature on the adhesive and cohesive strength of egg yolk deposits

For experiments on the adhesive strength of egg yolk deposits soaked in water for 15 minutes, increasing the hydration temperature from 20^{o}C to 40^{o}C reduced the adhesive strength of deposits by 0.275 % and increasing the hydration temperature from 20^{o}C to 60^{o}C reduced the adhesive strength by 3.21 %. For experiments on the adhesive strength of egg yolk samples soaked in 1 % w/v sodium hydroxide for 15 minutes, increasing the hydration temperature from 20^{o}C to 40^{o}C results in reduction of the adhesive strength by 6.43 %, while increasing the hydration temperature from 20^{o}C to 60^{o}C reduced the adhesive strength by 13.89 %. Increasing hydration temperature from 20^{o}C to 40^{o}C results in reduction of the adhesive strength of the egg yolk deposits hydrated in 1 % w/v LAS solution for 15 minutes by 3.72 %, while increasing the soaking temperature from 20^{o}C to 60^{o}C decreased the adhesive strength of deposits by 12.15 %. For experiments on the adhesive strength of egg yolk samples soaked in 5 % w/v sodium hydroxide for 15 minutes, increasing the soaking temperature from 20^{o}C to 40^{o}C results in reduction of the adhesive strength of deposits by 10.48 %, and increasing the soaking temperature from 20^{o}C to 60^{o}C decreased the adhesive strength of deposits by 13.89 %. Increasing hydration temperature from 20^{o}C to 40^{o}C results in reduction of the adhesive strength of the egg yolk deposits hydrated in 5 % w/v LAS solution for 15 minutes by 3.15 %, while increasing the hydration temperature from 20^{o}C to 60^{o}C reduced the adhesive strength of deposits by 15.51 %.

For experiments on the adhesive strength of egg yolk deposits soaked in water for 30 minutes, increasing the hydration temperature from 20^{o}C to 40^{o}C reduced the adhesive

strength of deposits by 13.95 % and increasing the hydration temperature from 20^{o}C to 60^{o}C reduced the adhesive strength by 44.79 %. Increasing the soaking temperature from 20^{o}C to 40^{o}C for samples soaked in 1 % w/v sodium hydroxide at 30 minutes hydration time, decreased the adhesive strength of deposits by 7.23 % while increasing the hydration temperature from 20^{o}C to 60^{o}C reduced the adhesive strength of the egg yolk deposits by 13.77 %. The adhesive strength of egg yolk samples hydrated in 1 % w/v LAS solution for 30 minutes was reduced by 3.26 % when the hydration temperature rose from 20^{o}C to 40^{o}C, while increasing the soaking temperature from 20^{o}C to 60^{o}C decreased the adhesive strength of the deposits by 24.32 %. The adhesive strength of egg yolk samples soaked in 5 % w/v sodium hydroxide for 30 minutes was decreased by 4.26 % when the hydration temperature increased from 20^{o}C to 40^{o}C, while raising the soaking temperature from 20^{o}C to 60^{o}C reduced the adhesive strength of the deposits by 14.37 %. For experiments on the adhesive strength of egg yolk deposits hydrated in 5 % w/v LAS solution for 30 minutes, the adhesive strength of deposits was decreased by 0.64 % when the hydration temperature increased from 20^{o}C to 40^{o}C while increasing the hydration temperature from 20^{o}C to 60^{o}C reduced the adhesive strength by 20.34 %.

The cohesive strength of egg yolk deposit soaked in water at 15 minutes hydration time was decreased by 5.52 % when the hydration temperature increased from 20^{o}C to 40^{o}C, while increasing the temperature from 20^{o}C to 60^{o}C reduced the cohesive strength of egg yolk deposit by 9.72 %. For samples soaked in 1 % w/v sodium hydroxide at 15 minutes hydration time, it was found that increasing the hydration temperature from 20^{o}C to 40^{o}C reduced the cohesive strength of the deposits by 7.86 %

while when the temperature was increased from 20^{o}C to 60^{o}C, the cohesive strength of the deposits was reduced by 16.62 %. The cohesive strength of samples hydrated in 1 % LAS solution at 15 minutes hydration time was also found to be reduced by 8.23 % when temperature was raised from 20^{o}C to 40^{o}C. When the temperature was increased from 20^{o}C to 60^{o}C, the cohesive strength of deposits was found to be reduced by 12.38 %. This similar trend was also found in the cohesive strength of deposits soaked in 5 % w/v sodium hydroxide and LAS solution at 15 minutes hydration where increasing the hydration temperature from 20^{o}C to 40^{o}C decreased the cohesive strength of deposits by 10.37 %, while increasing the hydration temperature from 20^{o}C to 60^{o}C reduced the cohesive strength of the deposits by 19.2 %.

For experiments on the cohesive strength of egg yolk deposits hydrated in water for 30 minutes, increasing the hydration temperature from 20^{o}C to 40^{o}C reduced the cohesive strength of deposits by 11.17 % and increasing the hydration temperature from 20^{o}C to 60^{o}C reduced the cohesive strength by 12.77 %. Increasing the soaking temperature from 20^{o}C to 40^{o}C for samples soaked in 1 % w/v sodium hydroxide at 30 minutes hydration time, decreased the cohesive strength of deposits by 6.57 % while increasing the hydration temperature from 20^{o}C to 60^{o}C reduced the cohesive strength of the egg yolk deposits by 14.49 %. The cohesive strength of egg yolk samples hydrated in 1 % w/v LAS solution for 30 minutes was reduced by 8.11 % when the hydration temperature rose from 20^{o}C to 40^{o}C, while increasing the soaking temperature from 20^{o}C to 60^{o}C decreased the cohesive strength of the deposits by 12.78 %. The cohesive strength of egg yolk samples soaked in 5 % w/v sodium hydroxide for 30 minutes was decreased by 7.12 % when the hydration temperature increased from

20°C to 40°C, while raising the soaking temperature from 20°C to 60°C reduced the cohesive strength of the deposits by 14.23 %. For experiments on the cohesive strength of egg yolk deposits hydrated in 5 % w/v LAS solution for 30 minutes, the cohesive strength of deposits was decreased by 11.56 % when the hydration temperature increased from 20°C to 40°C while increasing the hydration temperature from 20°C to 60°C reduced the cohesive strength by 16.28 %.

Liu *et al.* (2002) reported that 90% reduction of the adhesive strength of baked tomato paste was observed after increasing the hydration temperature from 25°C to 50°C.

Liu *et a.l* (2006) stated that the adhesive strength of whey protein concentration (WPC) deposits pre-soaked in water, 0.1 %, 0.5 %, 1 %, 2 % and 5 % w/v sodium hydroxide was reduced 0.98 Jm^{-2}, 2.6 Jm^{-2}, 5.92 Jm^{-2}, 4.85 Jm^{-2}, 3.36 Jm^{-2}, and 3.43 Jm^{-2} respectively after increasing the hydration temperature from 20°C to 70°C. It was also understood that temperature controls the diffusion of chemical agent into the deposit as the diffusion coefficient is thermally activated, and any chemical reaction which decreases the deposit strength would also be thermally activated. Study by Liu et al. (2007) indicates that increasing the hydration temperature from 20°C to 70°C decreased the minimum adhesive strength of egg albumin deposits at different hydration time and time taken to reach the minimum adhesive strength of egg albumin deposits. At 20°C the minimum adhesive strength was 0.95 Jm-2, while at 50°C and 70°C, the minimum adhesive strength was 0.24 Jm^{-2} and 0.02 Jm-2. Rois Anwar (2016) found that increasing the temperature from 25°C to 35°C decreased almost 90 % (from 2000 Jm^{-2} to 250 Jm^{-2}) of the pulling energy of chocolate deposit fouled on a polycarbonate

surface. This agrees with the present study that increasing the hydration temperature reduced the pulling strength of the egg yolk deposits.

6.2.9 The effect of chemical concentration on the adhesive and cohesive strength of egg yolk deposit

Experiments were conducted to study the effect of different chemical concentration on the adhesive and cohesive strength of egg yolk deposit. 0, 1 % and 5 % w/v sodium hydroxide and LAS solution were used in this set of experiments. For samples soaked in sodium hydroxide at 20^{o}C and 15 minutes hydration, increasing the chemical concentration from 0 % to 5 % w/v decreased the adhesive strength by 27.55 % and 15.35 % of the cohesive strength of deposits. At 40^{o}C and 15 minutes hydration, raising the chemical concentration from 0 % to 5 % w/v reduced 34.95 % of the adhesive strength and 16.92 % of the cohesive strength of deposits. At 60^{o}C and 15 minutes pre-soaking, the deposits adhesive strength was reduced by 35.53 % and the reduction of deposits cohesive strength was 24.24 % when the chemical concentration increased from 0 % to 5 % w/v. For samples soaked in LAS solution at 20^{o}C and 15 minutes hydration, the reduction of adhesive strength and cohesive strength of egg yolk deposits was found to be 21.01 % and 5.67 % respectively when the chemical concentration increased from 0 % to 5 % w/v. At 40^{o}C and 15 minutes pre-soaking, increasing the chemical concentration from 0 % to 5 % w/v results in 23.29 % and 10.5 % reduction of the adhesive and cohesive strength of egg yolk deposits. At 60^{o}C, increasing the chemical concentration from 0 % to 5 % w/v decreased the adhesive and cohesive strength of egg yolk deposits by 31.05 % and 11.09 % respectively. For egg yolk deposits hydrated in sodium hydroxide at 20^{o}C and 30 minutes hydration, it was found

that 36.05 % of the deposits adhesive strength and 16.68 % of the deposits cohesive strength was decreased when the chemical concentration increased from 0 % to 5 % w/v. At 40^{o}C and 30 minutes hydration, increasing the chemical concentration 0 % to 5 % w/v reduced the adhesive and cohesive strength of deposits by 28.85 % and 12.87 % respectively. At 60^{o}C and 30 minutes of hydration, the adhesive strength and cohesive strength of deposits was reduced by 30.55 % and 18.01 % respectively when the chemical concentration increased from 0 % to 5 % w/v. For samples soaked in LAS solution at 20^{o}C and 30 minutes pre-soaking, the reduction of adhesive strength and cohesive strength of samples was found to be 24.85 % and 8.05 % respectively when the chemical concentration increased from 0 % to 5 % w/v. At 40^{o}C and 15 minutes pre-soaking, increasing the chemical concentration from 0 % to 5 % w/v results in 13.24 % and 8.4 % reduction of the adhesive and cohesive strength of egg yolk deposits. At 60^{o}C, increasing the chemical concentration from 0 % to 5 % w/v decreased the adhesive and cohesive strength of egg yolk deposits by 24.08 % and 11.69 % respectively.

Liu et al 2006 reported that increasing the chemical concentration provides greater driving force for chemical diffusion and increase the reaction rate. Therefore it was found that increasing the chemical concentration reduced the pulling strength of the whey protein concentration (WPC). Increasing the chemical concentration from 0 to 0.1, 0.5, 1, 2, and 5 % w/v sodium hydroxide reduced the pulling strength of the whey protein concentration by 0.25, 0.82, 2.27, 4.01 and 4.09 Jm^{-2}. Liu et al. (2007) stated that the chemical reaction which involves cleaning chemical weakens the intermolecular forces, thus creates greater driving force for chemical diffusion and higher reaction rate.

They also reported that chemical processes dominates during the diffusion and swelling phase where the cleaning agent has to be transported through the deposit and molecular bonds have to be broken down. The apparent adhesive strength of egg albumin deposits was reduced from 2.94 to 0.69 Jm^{-2} when the concentration of sodium hydroxide was increased from 0 to 2 %. No significant reduction of adhesive strength of egg albumin deposits was found when the chemical concentration increased above 2 %. Rois Anwar (2016) found that increasing the sodium hydroxide concentration from 0 to 0.1 % w/v reduced the pulling strength of chocolate deposits and dissolution phenomena occurred when chemical was introduced as the pre-soaking solution. This is aligned with the results from the current study that increasing the chemical concentration decreased the pulling strength of egg yolk deposits.

6.2.10 The effect of hydration time on the adhesive and cohesive strength of egg yolk deposit

This set of experiments focus on the effect of hydration time on the adhesive and cohesive strength of egg yolk deposit using 15 minutes and 30 minutes hydration time. For samples soaked in sodium hydroxide at 1 % w/v concentration and 20^{o}C, increasing the hydration time from 15 minutes to 30 minutes decreased 17.99 % of the adhesive strength and 4.63 % of the cohesive strength of egg yolk deposit. At 40^{o}C and 1 % w/v concentration, the reduction of adhesive strength and cohesive strength of the deposits was found to be 18.69 % and 3.3 % respectively when the hydration time increased from 15 minutes to 30 minutes. At 60^{o}C and 1 % w/v concentration, the adhesive strength and cohesive strength of egg yolk deposits was decreased by 17.9 % and 2.2

% respectively when the hydration time increased from 15 minutes to 30 minutes. For samples hydrated in sodium hydroxide at 20^{o}C and 5 % w/v concentration, increasing the hydration time increased from 15 minutes to 30 minutes reduced the adhesive strength by 14.29 % and 5.67 % of the cohesive strength of deposits. At 40^{o}C and 5 % w/v concentration, raising the hydration time from 15 minutes to 30 minutes decreased 25.01 % of the adhesive strength and 5.51 % of the cohesive strength of deposits. At 60^{o}C and 5 % w/v concentration, the deposits adhesive strength was reduced by 26.5 % and the reduction of deposits cohesive strength was 9.79 % when the hydration time increased from 15 minutes to 30 minutes. For samples soaked in LAS solution at 20^{o}C and 1 % w/v concentration, the reduction of adhesive strength and cohesive strength of egg yolk deposits was found to be 5.44 % and 7.66 % respectively when the hydration time increased from 15 minutes to 30 minutes. At 40^{o}C and 1 % w/v concentration, increasing the hydration time increased from 15 minutes to 30 minutes results in 4.99 % and 7.54 % reduction of the adhesive and cohesive strength of egg yolk deposits. At 60^{o}C and 1 % w/v concentration, increasing the hydration time increased from 15 minutes to 30 minutes reduced the adhesive and cohesive strength of egg yolk deposits by 18.54 % and 8.09 % respectively. For egg yolk deposits hydrated in LAS solution at 20^{o}C and 5 % w/v concentration, it was found that 7.61 % of the deposits adhesive strength and 10.1 % of the deposits cohesive strength was decreased when the hydration time increased from 15 minutes to 30 minutes. At 40^{o}C and 5 % w/v concentration, increasing the hydration time increased from 15 minutes to 30minutes reduced the adhesive and cohesive strength of deposits by 5.22 % and 7.78 % respectively. At 60^{o}C and 5 % w/v concentration, the adhesive strength and cohesive

strength of deposits was reduced by 12.89 % and 8.02 % respectively when the chemical concentration increased from 1 % to 5 % w/v. For samples soaked in water at 20^{o}C, the hydration time increased from 15 minutes to 30minutes the reduction of adhesive strength and cohesive strength of samples was found to be 2.88 % and 4.16 % respectively when the hydration time increased from 15 minutes to 30minutes. At 40^{o}C, increasing the hydration time from 15 minutes to 30 minutes results in 16.19 % and 9.88 % reduction of the adhesive and cohesive strength of egg yolk deposits. At 60^{o}C, increasing the hydration time increased from 15 minutes to 30minutes decreased the adhesive and cohesive strength of egg yolk deposits by 20.87 % and 7.4 % respectively.

Liu et al. (2002) found that increasing hydration time from 5 minutes to 30 minutes decreased the apparent adhesive strength of tomato paste deposits by a factor of about 3 (7 Jm^{-2} down to 2 Jm^{-2}). The apparent adhesive strength remained constant after 30 minutes of hydration. Study by Liu et al. (2006) also found that the apparent adhesive strength of WPC was decreased when the hydration time was increased. At 20^{o}C, the reduction rate of the adhesive strength was about 0.1 $Jm^{-2}min^{-1}$ over a period of 50 minutes and the adhesive strength remained constant after 50 minutes of hydration while at 50^{o}C, the adhesive strength reduced at 0.3 $Jm^{-2}min^{-1}$ over 30 minutes of hydration and it remained unchanged after 30 minutes of hydration. At 70^{o}C, the reduction of the adhesive strength was about 1.2 $Jm^{-2}min^{-1}$ over a period of 5 minutes and remained constant after 5 minutes hydration time. Liu et al. (2007) also reported that increase in hydration time reduced the apparent adhesive strength of egg albumin deposits. At 20^{o}C, the apparent adhesive strength reduced to the minimum strength

(0.95 Jm^{-2}) after 60 minutes hydration while at 50^{o}C and 70^{o}C the apparent adhesive strength decreased to the minimum strength (0.02 Jm^{-2} and 0.24 Jm^{-2} respectively) after 45 minutes and 30 minutes respectively. Rois Anwar (2016) found an interesting behavior after 10 minutes of hydration where the pulling energy of chocolate deposits increased from 600 Jm-2 to 2000 Jm-2 between 7 to 10 minutes hydration and there was a slight increase in pulling energy at 20 minutes hydration time, while at 30 minutes soaking time, the pulling energy was reduced to 1800 Jm-2. This agrees with the results from the present study that pulling strength of egg yolk deposits is a function of the hydration time, where increasing the hydration time reduced the pulling strength of egg yolk deposits.

6.2.11 Adhesive strength vs cohesive strength

Apparently, it was found that adhesive strength of deposits is higher than cohesive strength of deposits in all cases (figure 6.57, 6.58, 6.59 and 6.60). This suggests that the force required to remove the egg yolk deposit completely from the stainless steel surface is higher than the force required to deform the deposit and to break the cohesive bonds between the deposits. Liu et al. (2002) found that the cohesive strength of tomato paste deposits is higher than the adhesive strength of tomato paste deposits. This is different to the current study on the removal of egg yolk deposits. Study by Liu et al. (2006) indicates that the adhesive strength of whey protein concentrate (WPC) is greater than the cohesive strength of WPC which agrees with the present study on the adhesive and cohesive strength of egg yolk deposits. Liu et al. (2007) reported that the cohesive strength of the egg albumin deposits is greater than the adhesive strength of egg albumin deposits. Again, this is different to the current study on the removal of egg

yolk deposits. Research by Cole (2013) found that more pulling energy was required to remove the toothpaste deposits at 0.1 mm gap between the probe and the surface (61.5 ± 2.6 Jm^{-2}) than pulling energy to remove toothpaste deposits at 1 mm gap (44.6 ± 1.2 Jm^{-2}), thus indicates that the adhesive strength of toothpaste deposits was greater than the cohesive strength of toothpaste deposits. This is aligned with the present study on the removal of egg yolk deposits. These findings suggest that the micromanipulation technique can be used to identify the different mechanical properties of different fouling species which will have an implication for effective cleaning of various fouling materials (Liu et al., 2006).

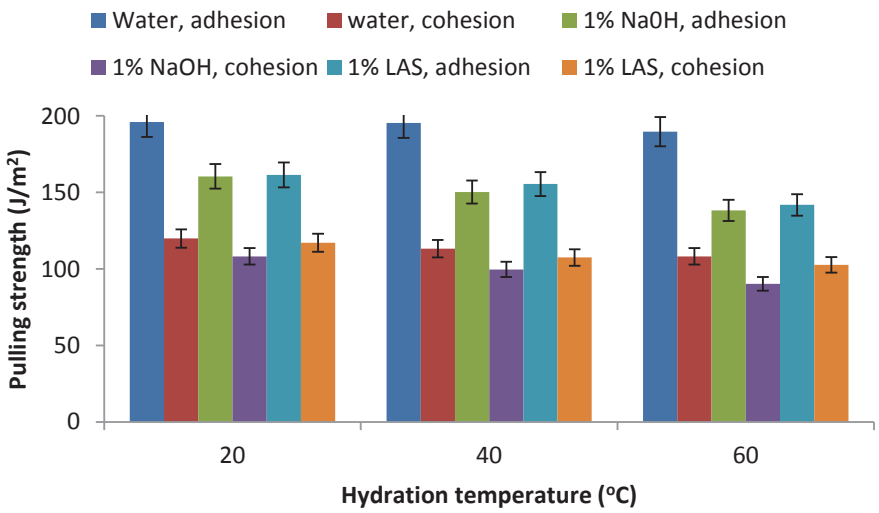


Figure 6.57: The adhesive and cohesive strength of egg yolk deposits soaked at 1 % w/v chemical concentration and 15 minutes hydration time

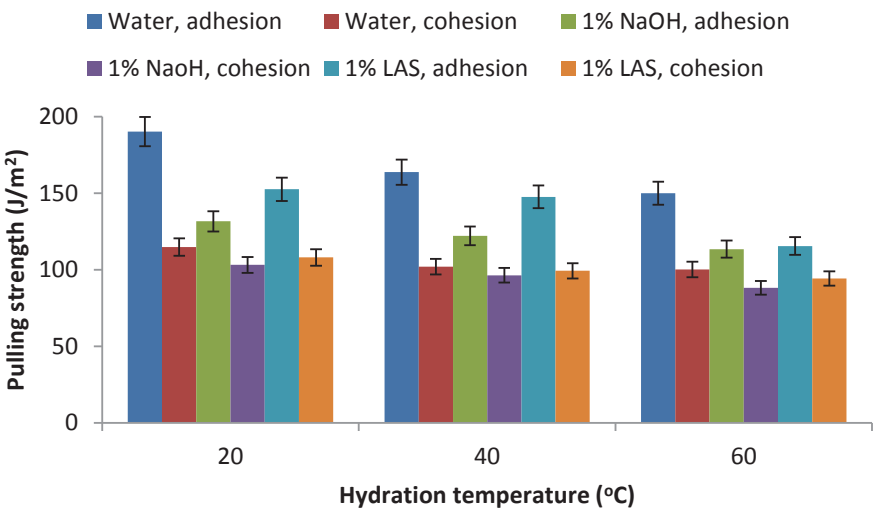


Figure 6.58: The adhesive and cohesive strength of egg yolk deposits soaked at 1 % w/v chemical concentration and 30 minutes hydration time

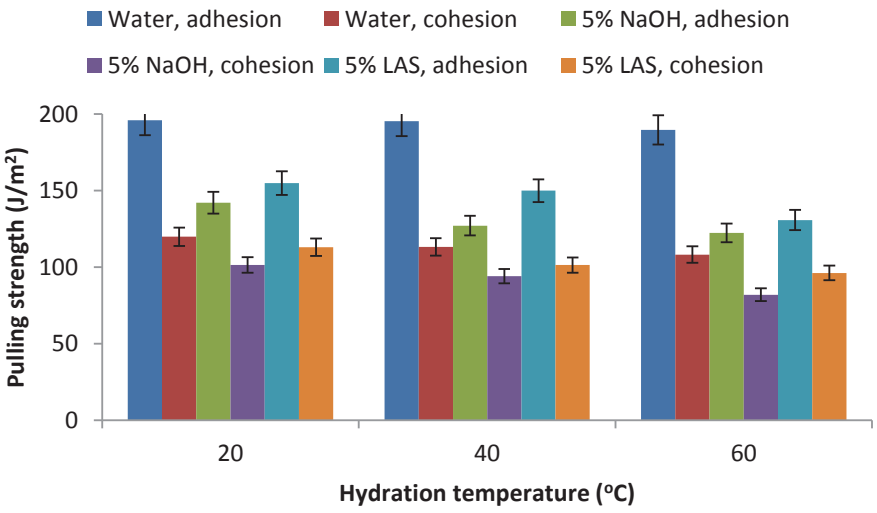


Figure 6.59: The adhesive and cohesive strength of egg yolk deposits soaked at 5 % w/v chemical concentration and 15 minutes hydration time

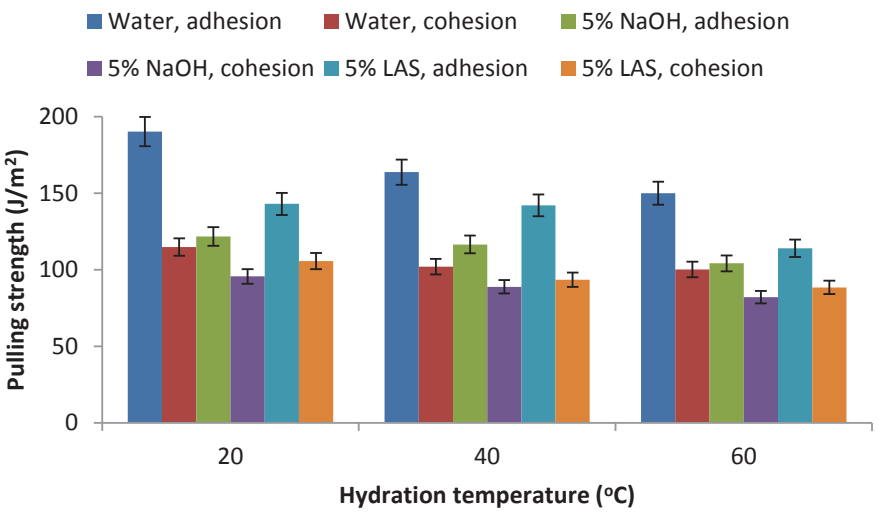


Figure 6.60: The adhesive and cohesive strength of egg yolk deposits soaked at 5 % w/v chemical concentration and 30 minutes hydration time

6.2.12 Novelty and innovation

Egg yolk powder is used to prepare the deposit samples in order to study the cleaning of a fat based fouling deposit using the cleaning rig and to measure the force needed to remove egg yolk deposit from a stainless steel coupon using micromanipulator. It is well understood that the previous researchers used similar method to study the swelling and the cleaning of a protein based deposit such as albumin, whey protein and other type 1 and type 2 fouling materials. Image analysis technique is introduced in this research to study and observe the cleaning progress. The image of the sample is snapped for every 5 minutes throughout the experiment and the collected images is analyzed using software called ImageJ. It is found that both temperature and chemical concentration are the key factor in the cleaning of egg yolk deposits. No complete removal found in the experiment at 40^{o}C. All experiments at 60oC and 80oC show total removal of egg yolk deposits with higher chemical concentration gives shorter cleaning time. Pre-

soaking temperature and chemical concentration really affect the adhesive and cohesive strength of egg yolk deposit in all cases, while some of the cases are not affected by longer pre-soaking time.

6.3 Conclusions

This cleaning rig can be used to study cleaning of egg yolk deposits fouled on stainless steel surface at laboratory scale. Cleaning was found to be divided into 3 phases i.e. lag phase, removal phase and constant phase. The heat transfer coefficient (U) profile and deposit resistance (R_d) profile from MHFS data can be used to determine cleaning time of egg yolk deposits. Cleaning of egg yolk deposits also can be observed and monitored using visual technique. No complete deposit removal was found at 40^{o}C in all cases. No complete deposit removal was achieved by cleaning with water even at the highest temperature (80^{o}C). At 60^{o}C and 80^{o}C, deposits were totally removed in all cases. Cleaning of egg yolk deposit was controlled by temperature and chemical concentration with cleaning of egg yolk deposits using 5 % w/v sodium hydroxide solution at 80^{o}C gave the shortest cleaning time (22 minutes) and cleaning using 1 % w/v LAS solution at 60^{o}C was found to have the longest cleaning time (172 minutes). Increasing the cleaning temperature and the chemical concentration of cleaning agent reduced the cleaning time and cleaning with sodium hydroxide solution gave shorter cleaning time than cleaning with LAS solution in all cases.

The micromanipulation technique can be used to determine the force required to remove egg yolk deposits from stainless steel surface. The adhesive strength of deposits were measured at the lowest gap (10 μm) between the probe and the surface

to make sure that there is no deposit layer left after experiment, while the cohesive strength of deposits was measured by adjusting the gap between the probe and the surface to 1 mm which leaves a layer of deposit on the surface after experiment done. The adhesive and cohesive strength of egg yolk deposits were a function of pre-soaking time, pre-soaking temperature and chemical concentration of pre-soaking solution. Increasing the chemical concentration of hydrating solution and the hydration time results in lower adhesive and cohesive strength of deposits while increasing the hydration temperature does not really affect the adhesive and cohesive strength of egg yolk deposit in some cases (based on statistical analysis). The adhesive strength of egg yolk deposits was found to be greater than the cohesive strength in all cases.

CHAPTER 7: CONCLUSIONS AND RECCOMENDATIONS

7.1 Conclusions

7.1.1 Swelling and removal of egg yolk deposits at different hydration temperature

The hydration experiment set up could be a useful method to study swelling and deposit removal of egg yolk. Temperature plays a major role in swelling and removal of egg yolk during hydration experiment. Higher temperature results in higher swelling performance and deposit removal. Increasing the hydration temperature from 40^{o}C to 60^{o}C helps to increase the swelling performance of egg yolk deposits. However, the swelling performance of egg yolk deposits soaked in 80^{o}C hydration solution did not increase as the swelling and deposit removal happens simultaneously during hydration. It is also found that temperature is a key factor in deposit removal. Samples soaked in higher hydration temperature gives higher deposit removal than samples soaked in lower hydration temperature. All samples soaked at 80^{o}C are totally removed after 120 minutes of experiment.

7.1.2 Chemical and mechanical action on swelling and cleaning of egg yolk deposit at room temperature

7.1.2.1 Chemical action

It is well understood that swelling of deposit is related to moisture content of deposit. Increase in moisture content leads to increase in swelling of deposit. Sodium hydroxide solution, LAS solution, and a commercial detergent solution are used in this study to investigate the effect of chemical action on the swelling and cleaning of egg yolk deposit

during hydration. From the results obtained, it is found that chemical action increases the swelling performance of egg yolk deposits with sodium hydroxide giving the highest swelling performance of egg yolk deposits. All samples soaked in all cleaning chemicals have higher swelling performance compares to those samples soaked in water. Chemical action helps to break down the chemical bonding between the deposit materials which leads to increase in moisture content and swelling of the deposits. Increase in swelling leads to increase in deposit removal. Sodium hydroxide solution appears to be the best chemical used to clean the egg yolk deposit which gives 55.01 % increase in deposit removal compares to those samples soaked in water, while LAS solution and detergent solution helps to raise the deposit removal by 40.75 % and 5.02 % respectively. This is aligned with the previous workers findings that chemical action really affects the removal of deposits.

7.1.2.2 Mechanical action

Magnetic stirrer and mixing impeller are used in this research to investigate the effect of mechanical action on the swelling and the cleaning of egg yolk deposit during hydration. Magnetic stirrer provides mechanical action to the system by creating vortex while mixing impeller provides mechanical action by creating axial flow. Results from the experiments indicate that both type of stirring help to improve the swelling performance of egg yolk deposits by up to 38.29 % without chemical action. It is also found that increase in swelling performance leads to higher deposit removal. The deposit removal is seen to be raised up to 38.33 % without any chemical actions. Therefore, it can be concluded that these results agree with the previous researches which stated that mechanical action plays an important role in the cleaning of food fouling deposit. It is

learned that Swelling and deposit removal occurs simultaneously in some cases. Chemical action is a main factor during swelling phase as it needs more chemical action to break down the deposits while mechanical action is needed to provide force to remove the deposit from the stainless steel surface.

7.1.3 Cleaning of egg yolk deposits at laboratory scale by flow cell rig

It is found that this cleaning rig can be an excellent experimental set up to study the cleaning of egg yolk deposit at laboratory scale. This experimental set up provides set of data for heat transfer coefficient profile and image analysis which are used to study the cleaning progress. It is understood that cleaning is divided into 3 phases i.e. lag phase, removal phase and constant phase. No complete deposit removal was found at 40^{o}C in all cases. At this temperature, chemical concentration plays a greater role which increases the deposit removal after 4 hours of rinsing. There is no complete removal for those samples clean using water even at the highest temperature (80^{o}C). However, cleaning with chemical (sodium hydroxide and LAS) at 60^{o}C and 80^{o}C show a total removal after 4 hours of experiment. It can be ideally suggested that the cleaning of egg yolk deposit is controlled by both temperature and chemical action with chemical action happens to provide higher effect than temperature. Increasing the temperature and the chemical concentration helps to reduce cleaning time.

7.1.4 The adhesive and cohesive strength of egg yolk deposits

It is well understood that the micromanipulation technique can be used to investigate the force needed to remove a food fouling deposit on a stainless steel surface. The gap

between the probe and the surface is adjusted to the lowest gap (100 μm) to ensure that no deposit layer left after scrapping in order to study the adhesive strength of deposit, while the cohesive strength of deposits was measured by adjusting the gap between the probe and the surface to 1 mm which leaves a layer of deposit on the surface after scrapping. Pre-soaking is a main factor in this experiments. No deposit removal was found without pre-soaking treatment. The adhesive and cohesive strength of egg yolk deposits are affected by pre-soaking time and chemical concentration of pre-soaking solution. Increasing the pre-soaking chemical concentration and hydration time gives lower adhesive and cohesive strength of deposits, while some of the cases show that the pre-soaking temperature does not really affect the adhesive and cohesive strength of egg yolk deposits. The adhesive strength of egg yolk deposits was found to be greater than the cohesive strength in all cases. Therefore, it can be suggested that egg yolk deposits removal is a function of cohesive failure.

7.2 Future work recommendations

7.2.1 Swelling and deposit removal during hydration experiment

It might be worth to have a proper set up for the hydration experiment. An improved system need to be introduced in order to derive more accurate data. The design should have its own thermo regulator so that a constant temperature can be obtained throughout the experiment. The set –up should have a stage to place the coupon (sample) so that the sample placed at the same position in every experiment carried out. In terms of experimental design, the effect of different hydration pH on swelling of egg yolk deposits should be investigated in future works. Perez-Mohedano et al. (2015)

found that pH has a major effect on swelling of egg yolk deposit and Mercade-Prieto et al (2007) reported that it is important to identify a pH threshold, suppressing pH and optimum pH for swelling of deposit. They also suggested that swelling of deposit would not take place at pH below pH threshold and at pH above suppressing pH. Therefore, it is crucial to investigate the pH threshold and the suppressing pH for swelling of egg yolk deposit so that the optimum pH for swelling of egg yolk deposit can be discovered. It might also be interesting to compare swelling of egg yolk deposits with other fat/protein based deposits.

7.2.2 Cleaning of egg yolk deposits by flow cell rig

The design of the cleaning rig should be modify or improve in order to get more accurate data. The computer used to collect data via data logger should be replaced with a new computer to ensure that the experiment can be carried out is efficient. The test section of the cleaning rig should be made easy for the user to place and remove the sample and also to prevent any leaking during experiment. The heater tank also needs to replace with a new one to ensure that it works properly during experiment. An improved image analysis technique should be designed in order to get more accurate data from the experiment. In terms of experimental design, It might be useful to look at cleaning of egg yolk deposits at different cleaning chemical pH, and at higher flow rate (more than 0.5 ms^{-1}), and compare the cleaning of egg yolk deposits with other fat/protein based deposits.

7.2.3 Measurement of force required to remove deposit by micromanipulation technique

As for the experimental set up, the computer used for the micromanipulator needs to be replaced with a new one because the current computer operates using window 95 and data obtained from the experiment cannot be transferred using USB terminal. It is worth to have new software for the micromanipulator so that the experiment can be carried out efficiently and the data obtained from the experiment can be easily transferred to other device. Apart from this, the stage where the sample is placed also needs to be more stable in order to have more accurate data. In terms of experimental design, It might be interesting to investigate the effect of different hydration pH on the adhesive and cohesive strength of egg yolk deposits and might be worth to compare the adhesive and cohesive strength of egg yolk deposits with other fat/protein based deposits.

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